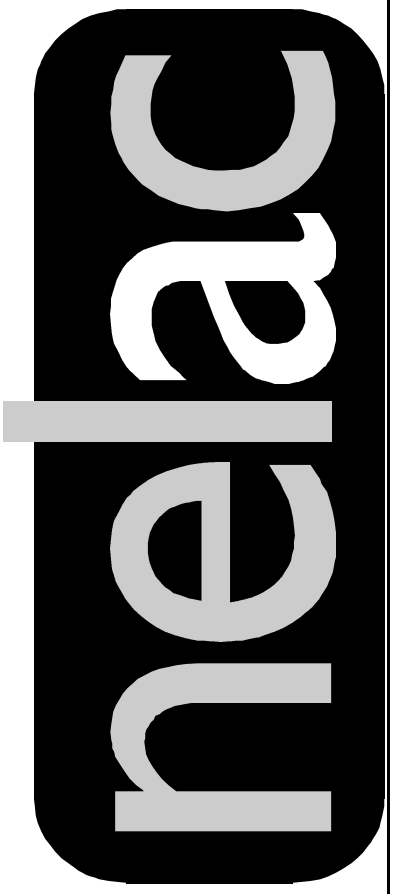
National Environmental Laboratory **Accreditation** Conference



QUALITY SYSTEMS

PROPOSED

April 29, 1999

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QUALITY SYSTEMS

NOTE: ***Additions (double-underlined) and ~~deletions~~ (struck through) to the approved standards being proposed for vote at the Fifth Annual Meeting are marked as in this note.***

5.0 QUALITY SYSTEMS

INTRODUCTION

Quality Systems include all quality assurance (QA) policies and quality control (QC) procedures, which shall be delineated in a Quality Manual and followed to ensure and document the quality of the analytical data. Laboratories seeking accreditation under NELAP must assure implementation of all QA policies and the essential applicable QC procedures specified in this chapter. The QA policies, which establish essential QC procedures, are applicable to environmental laboratories regardless of size and complexity.

The intent of this Chapter is to provide sufficient detail concerning quality management requirements so that all accrediting authorities evaluate laboratories consistently and uniformly.

NELAC is committed to the use of Performance Based Measurement Systems (PBMS) in environmental testing and provides the foundation for PBMS implementation in these standards. While this standard may not currently satisfy all the anticipated needs of PBMS, NELAC will address future needs within the context of state statutory and regulatory requirements and the finalized EPA implementation plans for PBMS.

Chapter 5 is organized according to the structure of ISO/IEC Guide 25, 1990. Where deemed necessary, specific areas within this Chapter may contain more information than specified by ISO/IEC Guide 25.

All items identified in this chapter shall be available for on-site inspection or data audit.

5.1 SCOPE

a) This Standard sets out the general requirements in accordance with which a laboratory has to demonstrate that it operates, if it is to be recognized as competent to carry out specific environmental tests.

b) This ~~s~~Standard includes additional requirements and information for assessing competence or for determining compliance by the organization or accrediting authority granting the recognition (or approval).

If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed (See the supplemental accreditation requirements in Section 1.~~8~~9.2.)

c) This Standard is for use by environmental testing laboratories in the development and implementation of their quality systems. It shall be used by accreditation authorities, in assessing the competence of environmental laboratories.

5.2 REFERENCES

See Appendix A

5.3 DEFINITIONS

The relevant definitions from ISO/IEC Guide 2, ISO 8402, ANSI/ASQC E-4,1994, the EPA "Glossary of Quality Assurance Terms and Acronyms", and the *International vocabulary of basic and general terms in metrology (VIM*) are applicable, the most relevant being quoted in Appendix B together with further definitions applicable for the purposes of this Standard.

See Appendix B

5.4 ORGANIZATION AND MANAGEMENT

5.4.1 Legal Definition of Laboratory

The laboratory shall be legally identifiable. It shall be organized and shall operate in such a way that its permanent, temporary and mobile facilities meet the requirements of this Standard**.**

5.4.2 Organization

The laboratory shall:

a) have managerial staff with the authority and resources needed to discharge their duties;

b) have processes to ensure that its personnel are free from any commercial, financial and other undue ~~-~~pressures which might adversely affect the quality of their work;

c) be organized in such a way that confidence in its independence of judgment and integrity is maintained at all times;

d) specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests;

Such documentation shall include:

1) a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured and

2) job descriptions for all positions.

e) provide supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test and the assessment of the results. The ratio of supervisory to non-supervisory personnel shall be such as to ensure adequate supervision~~;~~ to ensure close adherence to laboratory procedures and accepted techniques.

f) have a technical director(s) (however named) who has overall responsibility for the technical operation of the environmental testing laboratory;

The technical director(s) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.

The technical director(s) shall meet the requirements specified in the Accreditation Process. (see 4.1.1.1)

g) have a quality assurance officer (however named) who has responsibility for the quality system and its implementation. The quality assurance officer shall have direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the technical director. Where staffing is limited, the quality assurance officer may also be the technical director or deputy technical director;

The quality assurance officer (and/or his/her designees) shall:

1) serve as the focal point for QA/QC and be responsible for the oversight and/or review of quality control data;

2) have functions independent from laboratory operations for which they have quality assurance oversight;

3) be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence;

4) have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC;

5) have a general knowledge of the analytical test methods for which data review is performed;

6) arrange for or conduct internal audits on the entire technical operation annually; and

7) notify laboratory management of deficiencies in the quality system and monitor corrective action.

h) nominate deputies in case of absence of the technical director(s) and/or quality assurance officer;

i) have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights (this may not apply to in-house laboratories);

j) when available, participate in inter-laboratory comparisons and proficiency testing programs. For purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter 2.0.

5.5 QUALITY SYSTEM - ESTABLISHMENT, AUDITS, ESSENTIAL QUALITY CONTROLS AND DATA VERIFICATION

5.5.1 Establishment

The laboratory shall establish and maintain a quality system based on the required elements contained in this chapter and appropriate to the type, range and volume of environmental testing activities it undertakes.

a) The elements of this quality system shall be documented in the organization’s quality manual.

b) The quality documentation shall be available for use by the laboratory personnel.

c) The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.

d) The laboratory management shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood, and implemented by all laboratory personnel concerned.

e) The quality manual shall be maintained current under the responsibility of the quality assurance officer.

5.5.2 Quality Manual

The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this Standard.

The Quality Manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality assurance officer (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;

The quality manual and related quality documentation shall also contain:

a) a quality policy statement, including objectives and commitments, by top management;

b) the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;

c) the relationship between management, technical operations, support services and the quality system;

d) procedures to ensure that all records required under this Chapter are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;

e) job descriptions of key staff and reference to the job descriptions of other staff;

f) identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the QA officer(s), technical director(s), and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager;

g) the laboratory's procedures for achieving traceability of measurements;

h) a list of all test methods under which the laboratory performs its accredited testing;

i) mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;

j) reference to the calibration and/or verification test procedures used;

k) procedures for handling submitted samples;

l) reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;

m) reference to procedures for calibration, verification and maintenance of equipment;

n) reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;

o) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

p) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;

q) procedures for dealing with complaints;

r) procedures for protecting confidentiality (including national security concerns), and proprietary rights;

s) procedures for audits and data review;

t) processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out ~~and/or receive~~and are receiving any needed training;

u) reference to procedures for reporting analytical results; and

v) a Table of Contents, and applicable lists of references and glossaries, and appendices.

5.5.3 Audits

5.5.3.1 Internal Audits

The laboratory shall arrange for annual internal audits to verify that its operations continue to comply with the requirements of the laboratory's quality system. It is the responsibility of the quality assurance officer to plan and organize audits as required by a predetermined schedule and requested by management. Such audits shall be carried out by ~~the quality assurance officer or designee(s) who are~~ trained and qualified ~~as auditors, and~~personnel who are, wherever ~~possible~~resources permit, independent of the activity to be audited. Personnel shall not audit their own activities except when it can be demonstrated that an effective audit will be carried out. Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work may have been affected.

5.5.3.2 Managerial Review

~~At least once per year, t~~The laboratory management shall conduct a review, at least annually, of its quality system and its testing and calibration activities to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements in the quality system and laboratory operations. The review shall take account of reports from managerial and supervisorial personnel, the outcome of recent internal audits, assessments by external bodies, the results of interlaboratory comparisons or proficiency tests, any changes in the volume and type of work

undertaken, feedback from clients, corrective actions and other relevant factors. The laboratory shall have a procedure for review by management and maintain records of review findings and actions.

5.5.3.3 Audit Review

All audit and review findings and any corrective actions that arise from them shall be documented. The laboratory management shall ensure that these actions are discharged within the agreed time frame .

5.5.3.4 Performance Audits

In addition to periodic audits, the laboratory shall ensure the quality of results provided to clients by implementing checks to monitor the quality of the laboratory's analytical activities. Examples of such checks are:

a) internal quality control procedures using whenever possible statistical techniques; (see 5.5.4 below)

b) participation in proficiency testing or other interlaboratory comparisons (See Chapter 2.0);

c) use of certified reference materials and/or in-house quality control using secondary reference materials as specified in Section 5.5.4;

d) replicate testings using the same or different test methods;

e) re-testing of retained samples;

f) correlation of results for different parameters of a sample (for example, total phosphorus should be greater than or equal to orthophosphate).

5.5.3.5 Corrective Actions

a) In addition to providing acceptance criteria and specific protocols for corrective actions in the Method Standard Operating Procedures (see 5.10.1.1), the laboratory shall implement general procedures to be followed to determine when departures from documented policies, procedures and quality control have occurred. These procedures shall include but are not limited to the following:

    1) identify the individual(s) responsible for assessing each QC data type;

    2) identify the individual(s) responsible for initiating and/or recommending corrective actions;

    3) define how the analyst should treat a data set if the associated QC measurements are unacceptable;

    4) specify how out-of-control situations and subsequent corrective actions are to be documented; and

    5) specify procedures for management (including the QA officer) to review corrective action reports.

b) To the extent possible, samples shall be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s).

5.5.4 Essential Quality Control Procedures

~~The following~~These general quality control principles shall apply, where applicable, to all testing laboratories. The manner in which they are implemented is dependent on the types of tests performed by the laboratory (i.e.~~g.~~, chemical, whole effluent toxicity, microbiological, radiological, air) and are further described in Appendix D. The standards for any given test type shall assure that the applicable principles are addressed:

a) All laboratories shall have protocols (as required in Section 5.10.1.1) in place to monitor the following quality controls:

1) Adequate positive and negative controls to monitor tests such as blanks, spikes, reference toxicants;

2) Adequate tests to define the variability and/or repeatability of the laboratory results such as replicates ;

3) Measures to assure the accuracy of the test method including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures;

4) Measures to evaluate test method capability, such as method detection limits and quantitation limits or range of applicability such as linearity;

5) Selection of appropriate formulae to reduce raw data to final results such as regression analysis, comparison to internal/external standard calculations, and statistical analyses ;

6) Selection and use of reagents and standards of appropriate quality;

7) Measures to assure the selectivity of the test for its intended purpose; and

8) Measures to assure constant and consistent test conditions (both instrumental and environmental) where required by the test method such as temperature, humidity, light, or specific instrument conditions.

b) All quality control measures shall be assessed and evaluated on an on-going basis, and quality control acceptance criteria shall be used to determine the ~~useability~~validity of the data (See Appendix D).

c) The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exist. (See 5.11.2, Sample Acceptance Policy.)

d) The quality control protocols specified by the laboratory's method manual (5.10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals

The essential quality control measures for testing are found in Appendix D of this chapter.

5.6 PERSONNEL

5.6.1 General Requirements for Laboratory Staff

The laboratory shall have sufficient personnel, having the necessary education, training, technical knowledge and experience for their assigned functions.

All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function. Each technical staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular function and a general knowledge of laboratory operations, ~~analytical~~ test methods, quality assurance/quality control procedures and records management.

5.6.2 Laboratory Management Responsibilities

In addition to 5.4.2.d, the laboratory management shall be responsible for:

a) Defining the minimal level of qualification, experience and skills necessary for all positions in the laboratory. In addition to education and/or experience, basic laboratory skills such as using a balance, colony counting, aseptic or quantitative techniques shall be considered;

b) Ensuring that all technical laboratory staff have demonstrated ~~initial and ongoing proficiency~~capability in the activities for which they are responsible. Such demonstration shall be documented (See Appendix C);

Note: In laboratories with specialized "work cells" (a group of well defined analyts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

c) Ensuring that the training of its personnel is kept up-to-date (on-going) by the following:

1) Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

2) Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.

3) Analyst training shall be considered up to date if an employee training file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

i. Acceptable performance of a blind sample (single blind to the analyst);

ii. Another ~~initial~~ demonstration of ~~method performance~~capability;

iii. Successful analysis of a blind performance sample on a similar test method using the same technology (e.g., GC/MS volatiles by purge and trap for 524.2, 624 or 5035/8260) would only require documentation for one of the test methods;

iv. At least four consecutive laboratory control samples with acceptable levels of precision and accuracy;

v. If ~~i~~i-iv cannot be performed, analysis of authentic samples that have been analyzed by another trained analyst with statistically ~~identical~~indistinguishable results.

d) Documenting all analytical and operational activities of the laboratory;

e) Supervising all personnel employed by the laboratory;

f) Ensuring that all sample acceptance criteria (Section 5.11) are verified and that samples are logged into the sample tracking system and properly labeled and stored; and

g) Documenting the quality of all data reported by the laboratory.

5.6.3 Records

Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory [see 5.6.2.c)], including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in 5.10.2.1 for chemical testing.

5.7 PHYSICAL FACILITIES - ACCOMMODATION AND ENVIRONMENT

5.7.1 Environment

a) Laboratory accommodation, test areas, energy sources, lighting, heating and ventilation shall be such as to facilitate proper performance of tests.

b) The environment in which these activities are undertaken shall not invalidate the results or adversely affect the required accuracy of measurement. Particular care shall be taken when such activities are undertaken at sites other than the permanent laboratory premises.

c) The laboratory shall provide for the effective monitoring, control and recording of environmental conditions as appropriate. Such environmental conditions may include biological sterility, dust, electromagnetic interference, humidity, mains voltage, temperature, and sound and vibration levels .

d) In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.

NOTE - It is the laboratory's responsibility to comply with the relevant health and safety requirements. This aspect, however, is outside the scope of this Standard.

5.7.2 Work Areas

a) There shall be effective separation between neighboring areas when the activities therein are incompatible including culture handling or incubation areas and volatile organic chemicals handling areas.

b) Access to and use of all areas affecting the quality of these activities shall be defined and controlled.

c) Adequate measures shall be taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality.

d) Work spaces must be available to ensure an unencumbered work area. Work areas include:

1) access and entryways to the laboratory;
2) sample receipt area(s);
3) sample storage area(s);
4) chemical and waste storage area(s); and
5) data handling and storage area(s).

5.8 EQUIPMENT AND REFERENCE MATERIALS

a) The laboratory shall be furnished with all items of equipment (including reference materials) required for the correct performance of tests for which accreditation is sought. In those cases where the laboratory needs to use equipment outside its permanent control it shall ensure that the relevant requirements of this Standard are met.

b) All equipment shall be properly maintained, inspected and cleaned. Maintenance procedures shall be documented.

c) Any item of the equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or otherwise to be defective, shall be taken out of service, clearly identified and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations or tests.

d) Each item of equipment including reference materials shall, when appropriate, be labeled, marked or otherwise identified to indicate its calibration status.

e) Records shall be maintained of each major item of equipment and all reference materials significant to the tests performed. These records shall include documentation on all routine and non-routine maintenance activities and reference material verifications.

The records shall include:

1) the name of the item of equipment;
2) the manufacturer's name, type identification, and serial number or other unique identification;
3) date received and date placed in service (if available);
4) current location, where appropriate;
5) if available, condition when received (e.g. new, used, reconditioned);
6) copy of the manufacturer's instructions, where available;
7) dates and results of calibrations and/or verifications and date of the next calibration and/or verification;
8) details of maintenance carried out to date and planned for the future; and
9) history of any damage, malfunction, modification or repair.

5.9 MEASUREMENT TRACEABILITY AND CALIBRATION

5.9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests shall be calibrated and/or verified before being put into service and on a continuing basis. The laboratory shall have an established program for the calibration and verification of its measuring and test equipment. This includes balances, thermometers and control standards.

5.9.2 Traceability of Calibration

a) The overall program of calibration and/or verification and validation of equipment shall be designed and operated so as to ensure that, wherever applicable, measurements made by the laboratory are traceable to national standards of measurement where available.

b) Calibration certificates ~~shall~~, when available, shall indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.

c) Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.

5.9.3 Reference Standards

a) Reference standards of measurement held by the laboratory (such as Class S or equivalent weights or traceable thermometers) shall be used for calibration only and for no other purpose, unless it can be demonstrated that their performance as reference standards have not been invalidated. Reference standards of measurement shall be calibrated by a body that can provide, where possible, traceability to a national standard of measurement.

b) There shall be a program of calibration and verification for reference standards.

c) Where relevant, reference standards and measuring and testing equipment shall be subjected to in-service checks between calibrations and verifications. Reference materials shall, where possible, be traceable to national or international standards of measurement, or to national or international standard reference materials.

~~**5.9.4 Calibration**~~

~~**5.9.4.1 General Requirements**~~

~~a) Each calibration shall be dated and labeled with or traceable to the test method, instrument, analysis date, and each analyte name, concentration and response (or response factor).~~

~~b) Sufficient information shall be recorded to permit reconstruction of the calibration.~~

~~c) Criteria for the acceptance of a calibration procedure, such as calibration curves and concentration (titer) determinations of titrants, shall be established. If applicable, the method specified criteria shall be met.~~

5.9.4.2 Acceptance Criteria for Support Equipment

5.9.4.2.1 Analytical Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All support equipment shall be:

a) maintained in proper working order. The records of all activities including service calls, shall be kept.

b) calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which is equipment is used or:

1) The equipment shall be removed from service until repaired; or

2) The laboratory shall prepare a deviation curve and correct all measurements for the deviation. All measurements shall be recorded and maintained.

c) Prior to use on each working day, balances, ovens, refrigerators, freezers, incubators and water baths shall be checked with NIST traceable references (where possible) in the expected use range. Additional monitoring as prescribed by the test method shall be performed for any device that is used in a critical test (such as incubators or water baths). The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used .

d) Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on a monthly use basis.

5.9.4.2.2 Autoclaves

The sterilization temperature and pressure of each run must be documented by the use of appropriate chemical or biological sterilization indicators. Autoclave tape may be used to indicate by color change that a load has been processed, but not to demonstrate completion of an acceptable sterilization cycle. Demonstration of sterilization may be provided by a continuous temperature recording or with the use of spore strips.

5.9.4.3 Instrument Calibrations

a) When available, all initial calibrations shall be verified with a standard obtained from a second or different source. This verification standard shall be analyzed with each initial calibration and shall be within 15% of the true value unless the laboratory can demonstrate through historical data that wider limits are applicable.

b) Calibration curves shall be prepared as specified in the test method. If a test method does not provide guidance in the preparation of a calibration curve, the laboratory shall establish the appropriate number of standards for use in the initial calibration using the following:

1) Determine the percent relative standard deviation (%RSD) by:

i. Taking at least seven replicate measurements of a standard with a concentration approaching the lowest quantitation level or;

ii. Performing a calibration linearity test (such as response factor or calibration factor) on at least 3 standards having concentrations that cover the expected calibration range.

2) The minimum number of standards to be used in the initial calibration is dependent on the resulting %RSD:

%RSD	Number of Calibration Points
0 - <2	1**
2 - <10	3
10 - <25	5
>25	7

** Assumes linearity through the origin (0.0). For analytes for which there is no origin (such as pH), a two point calibration curve shall be used.

3) If the resulting curve is non-linear, additional standards shall be used.

4) The number of standards as determined from the above table and a blank shall be used for the initial calibration of the test method.

c) In addition to the verification by second-source standards [see a) above], the calibration curve shall be subjected to a calibration linearity test, such as a linear regression or percent RSD of response factors (internal standard calibration) or calibration factors (external standard calibration).

1) If, over the calibration range, the RSD of response factors is less than 15 percent, or the RSD of calibration factors is less than 30 percent, linearity through the origin can be assumed and an average relative response factor may be used; otherwise, the complete calibration curve shall be used.

2) If a linear regression is used, the correlation coefficient (R) shall be no less than 0.995 unless the laboratory can demonstrate that a lowered correlation coefficient consistently produces accurate results.

d) For results to be reported as quantitative [i.e., those greater than 3.18 times the Method Detection Limit (MDL)] they must be bracketed by calibration or calibration verification standards. All other results must be reported as having a lower confidence level.

5.9.4.4 Calibration Verification

When not included in the analytical test method, the value of the analyte(s) in the following calibration verification standards shall be within 15% of the true value unless the laboratory can demonstrate through historical data that wider limits are applicable.

~~**5.9.4.4.1 Initial Calibration Verification**~~

~~a) When an initial calibration curve is not established on the day of analysis, the integrity of the initial calibration curve shall be verified on each day of use (or 24 hour period) by initially analyzing a blank and a standard at the method defined concentration or a mid-level concentration if not included in the test method.~~

~~b) If the initial calibration verification fails, the analysis procedure shall be stopped and evaluated. For example, a second standard may be analyzed and evaluated or a new initial calibration curve may be established and verified. In all cases, the initial calibration verification must be acceptable before analyzing any samples.~~

~~**5.9.4.4.2 Continuing Calibration Verification**~~

~~Additional standards shall be analyzed after the initial calibration curve or the integrity of the initial calibration curve (see 5.9.4.3.a or 5.9.4.4.1 above) has been accepted.~~

~~a) These standards shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent and may be the standards used in the original calibration curve or standards from another source. The frequency shall be increased if the instrument consistently drifts outside acceptance criteria before the next calibration.~~

~~b) The concentration of these standards shall be determined by the anticipated or known concentration of the samples and/or method specified levels. At least one standard shall be at a low level concentration. To the extent possible, the samples in each interval (i.e. every 20 samples or every 12 hours) should be bracketed with standard concentrations closely representing the lower and upper range of reported sample concentrations. If this is not possible, the standard calibration checks should vary in concentration throughout the range of the data being acquired.~~

~~c) If a calibration check standard fails, and routine corrective action procedures fail to produce a second consecutive calibration check within acceptance criteria, a new initial calibration curve shall be constructed. When the continuing calibration [check] acceptance criteria are exceeded high (i.e., high bias), and there are non-detects for the corresponding analyte in all environmental samples associated with the continuing calibration check, then those non-detects may be reported, otherwise the samples affected by the unacceptable check shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Additional sample analysis shall not occur until a new calibration curve is established and verified.~~

5.9.4 Calibration

Calibration requirements are divided into two parts: (1) requirements for analytical support equipment, and 2) requirements for instrument calibration. In addition, the requirements for instrument calibration are divided into initial instrument calibration and continuing instrument calibration verification.

5.9.4.1 Support Equipment

These standards apply to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors), thermal/pressure sample preparation devices and volumetric

dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All support equipment shall be:

a) Maintained in proper working order. The records of all repair and maintenance activities including service calls, shall be kept.

b) Calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

1) The equipment shall be removed from service until repaired; or

2) The laboratory shall maintain records of established correction factors to correct all measurements.

c) Raw data records shall be retained to document equipment performance.

d) Prior to use on each working day, balances, ovens, refrigerators, freezers, incubators and water baths shall be checked with NIST traceable references (where possible) in the expected use range. Additional monitoring as prescribed by the test method shall be performed for any device that is used in a critical test (such as incubators or water baths). The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

e) Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on at least a quarterly use basis. Glass microliter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory.

f) For chemical tests the temperature and pressure of each run of autoclaves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

g) For biological tests the sterilization temperature and pressure of each run of autoclaves must be documented by the use of appropriate chemical or biological sterilization indicators. Autoclave tape may be used to indicate by color change that a load has been processed, but not to demonstrate completion of an acceptable sterilization cycle. Demonstration of sterilization shall be provided by a continuous temperature recording with the frequent use of spore strips.

5.9.4.2 Instrument Calibrations

This standard specifies the essential elements that will define the procedures and documentation for initial instrument calibration and continuing instrument calibration verification to ensure that the data will be of known quality and be appropriate for a given regulation or decision. This standard does not specify detailed procedural steps ("how to") for calibration, but establishes the essential elements for selection of the appropriate technique(s). This approach allows flexibility and permits the employment of a wide variety of analytical procedures and statistical approaches currently applicable for calibration. If more stringent standards or requirements are included in a mandated test method or by regulation, the laboratory shall demonstrate that such requirements are met. If

it is not apparent which standard is more strigent, then the requirements of the regulation or mandated test method are to be followed.

Note: In the following sections, initial insturment calibration is directly used for quantitation and continuing instrument calibration verification is used to confirm the continued validity of the initial calibration.

5.9.4.2.1 Initial Instrument Calibration:

The following items are essential elements of initial instrument calibration:

a) The details of the initial instrument calibration procedures including calculations, integrations, and associated statistics must be included or referenced in the test method SOP.

b) Sufficient raw data records must be retained to permit reconstruction of the initial instrument calibration, e.g., calibration date, test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor.

c) Sample results must be quantitated from the initial instrument calibration and may not be quantitated from any continuing insturment calibration verification.

d) All initial instrument calibrations must be verified with a standard obtained from a second and traceable to a national standard, when available.

e) Criteria for the acceptance of an initial instrument calibration must be established, e.g., correlation coefficient and relative percent difference.

f) Results of samples not bracketed by initial instrument calibration standards (within calibration range) must be reported as having less certainty, e.g., defined qualifiers or flags or explained in the case narrative.

g) If the initial instrument calibration results are outside established acceptance criteria, corrective actions must be performed. Data associated with an unacceptable initial instrument calibration shall not be reported.

h) Calibration standards must include concentrations at or below the regulatory limit/decision level, if these limits/levels are known by dthe laboratory, unless these concentrations are below the laboratory's demonstrated detection limits (See D.1.4 Detection Limits)

i) The minimum number of concentration points for performing an initial instrument calibration is two. The laboratory must have a standard operating procedure for determining the number of points for establishing the initial instrument calibration.

5.9.4.2.2 Continuing Instrument Calibration Verification

When an initial instrument calibration is not performed on the day of analysis, the validity of the initial calibration shall be verified prior to sample analyses by a continuing instrument calibration verification with each analytical batch. The following items are essential elements of continuing instrument calibration verification:

a) The details of the continuing instrument calibration procedure, calculations and associated statistics must be included or referenced in the test method SOP.

b) A continuing instrument calibration verification must be repeated at the beginning and end of each analytical batch. The concentrations of the calibation verification shall be varied within the established calibration range. If an internal standard is used, only one continuing instrument calibration verification must be analyzed per analytical batch.

c) Sufficient raw data records must be retained to permit reconstruction of the continuing instrument calibration verification, e.g., test method, instrument, analysis date, each analyte name, concentration and response, calibration curve or response factor.

d) Criteria for the acceptance of a continuing instrument calibration verification must be established, e.g., relative percent difference.

e) If the continuing instrument calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, a new initial instrument calibration must be performed. Additional samples analyses shall not occur until a new calibration curve is established and verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

   i. When the acceptance criteria for the continuing calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

   ii. When the acceptance criteria for the continuing calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.

5.10 TEST METHODS AND STANDARD OPERATING PROCEDURES

5.10.1 Methods Documentation

a) The laboratory shall have documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.

b) All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.

5.10.1.1 Standard Operating Procedures (SOPs)

Laboratories shall maintain standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods.

a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents.

b) The test methods may be copies of published methods as long as any changes in the methods are documented and included in the methods manual (see 5.10.1.2).

c) Copies of all SOPs shall be accessible to all personnel.

d) The SOPs shall be organized .

e) Each SOP shall clearly indicate the effective date of the document, the revision number and the signature(s) of the approving authority.

5.10.1.2 Laboratory Method Manual(s)

a) The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.

b) This manual may consist of copies of published or referenced test methods or standard operating procedures that have been written by the laboratory. In cases where modifications to the published method have been made by the laboratory or where the referenced test method is ambiguous or provides insufficient detail, these changes or clarifications shall be clearly described. Each test method shall include or reference where applicable:

1) identification of the test method;
2) applicable matrix or matrices;
3) method detection limit;
4) scope and application, including components to be analyzed;
5) summary of the test method;
6) definitions;
7) interferences;
8) safety;
9) equipment and supplies;
10) reagents and standards;
11) sample collection, preservation, shipment and storage;
12) quality control;
13) calibration and standardization;
14) procedure;
15) calculations;
16) method performance;
17) pollution prevention;
18) data assessment and acceptance criteria for quality control measures;
19) corrective actions for out-of-control data;
20) contingencies for handling out-of-control or unacceptable data;
21) waste management;
22) references; and
23) any tables, diagrams, flowcharts and validation data

5.10.2 Test Methods

a) The laboratory shall use appropriate test methods and procedures for all tests and related activities within its responsibility (including sample collection, sample handling, transport and storage, sample preparation and sample analysis). The method and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations or tests concerned.

1) When the use of specific test methods for a sample analysis are mandated or requested, only those methods shall be used.

2) Where test methods are employed that are not required, as in the Performance Based Measurement System approach, the methods shall be fully documented and validated (see 5.10.2.1 and Appendix C), and be available to the client and other recipients of the relevant reports.

5.10.2.1 ~~Method Validation/Initial~~ Demonstration of Capability

a) Prior to acceptance and institution of any test method, satisfactory ~~initial~~ demonstration of method ~~performance is required~~ capability is required. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids, biological tissue and air. However, actual sample spike results may be used to meet this standard, i.e.,at least 4 consecutive matrix spikes within the last 12 months. In addition, for analytes which do not lead themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

1) The laboratory's use of mandated test methods [see 5.10.2.a)1] or EPA reference test methods, shall follow the protocols outlined in Appendix C of this document.

2) All other test methods (including Performance Based Measurements Systems) shall follow the protocols outlined in Appendix E of this document.

3) Exceptions to these requirements are ~~microbiology and~~ tests for which spiking ~~solutions are not available, for example, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color~~is not an option and for which Quality Control Samples are not readily available, such as microbiology, odor, temperature, and dissolved oxygen ~~or turbidity~~.

b) Thereafter, continuing demonstration of method performance, as per the quality control requirements in Appendix D,(such as laboratory control samples) is required.

c) In all cases, the appropriate forms such as the Certification Statement (Appendix C) or standard performance checklists (see Appendix E) must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the checklists must be retained by the laboratory.

d) ~~Initial~~A demonstration of ~~method performance~~capability must be completed each time there is a significant change in instrument type, personnel, or test method.

e) In laboratories with a specialized "work cell/s" (a group consisting of analysts with specifically defined tasks that together perform the test method), the group as a unit must meet the above criteria and this demonstration of capability must be fully documented.

f) When a work cell/s is employed, and the members of the cell change, the new employee/s must work with experienced analyst/s in the speciality area and this new work cell must demonstrate acceptable performance through acceptable continuing performance checks (appropriate sections of Appendix D, such as laboratory control samples). Such performance must be documented and the 4 preparation batches following the change in

personnel must not result in the failure of any batch acceptance criteria, e.g., method blank and laboratory control sample, or the demonstration of capability must be repeated. In addition, if the entire work cell is changed/replaced, the work cell must repeat the demonstration of capability (Appendix C).

g) When a work cell/s is employed the performance of the group must be linked to the training record of the individual members of the work cell (see section 5.6.2)

5.10.3 Sample Aliquots

Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.

5.10.4 Data Verification

Calculations and data transfers shall be subject to appropriate checks.

a) The laboratory shall establish Standard Operating Procedures to ensure that the reported data ~~is~~are free from transcription and calculation errors.

b) The laboratory shall establish a Standard Operating Procedures to ensure that all quality control measures are reviewed, and evaluated before data are reported.

5.10.5 Documentation and Labeling of Standards and Reagents

Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.

a) The laboratory shall retain records for all standards including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless it is verified by the laboratory.

b) Original containers (such as provided by the manufacturer or vendor) shall be labeled with an expiration date.

c) ~~Detailed r~~Records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

d) All containers of prepared reagents and standards must bear a unique identifier and expiration date and be linked to the documentation requirements in 5.10.5.c) above.

5.10.6 Computers and Electronic Data Related Requirements

Where computers or automated equipment are used for the capture, processing, manipulation, recording, reporting, storage or retrieval of test data, the laboratory shall ensure that:

a) all requirements of this Standard (i.e. Chapter 5) are complied with. Section 8.1 through 8.11 of the EPA Document "2185 - Good Automated Laboratory Practices" (1995), shall be

adopted as the standard for all laboratories employing microprocessors ~~and~~, computers, as well as, laboratories employing Laboratory Information Management Systems.

b) computer software is documented and adequate for use;

c) procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission and data processing;

d) computer and automated equipment are maintained to ensure proper functioning and provided with the environmental and operating conditions necessary to maintain the integrity of calibration and test data;

e) it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.

5.11 SAMPLE HANDLING, SAMPLE ACCEPTANCE POLICY AND SAMPLE RECEIPT

While the laboratory may not have control of field sampling activities, the following are essential to ensure the validity of the laboratory's data.

5.11.1 Sample Tracking

a) The laboratory shall have a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.

b) This laboratory code shall maintain an unequivocal link with the unique field ID code assigned each container.

c) The laboratory ID code shall be placed on the sample container as a durable label.

d) The laboratory ID code shall be entered into the laboratory records (see 5.11.3.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.

e) In cases where the sample collector and analyst are the same individual or the laboratory preassigns numbers to sample containers, the laboratory ID code may be the same as the field ID code.

5.11.2 Sample Acceptance Policy

The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples which do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:

a) Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;

b) Proper sample labeling to include unique identification and a labeling system for the samples with requirements concerning the durability of the labels (water resistant) and the use of indelible ink;

c) Use of appropriate sample containers;

d) Adherence to specified holding times;

e) Adequate sample volume. Sufficient sample volume must be available to perform the necessary tests; and

f) Procedures to be used when samples which show signs of damage or contamination.

5.11.3 Sample Receipt Protocols

a) Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, shall be recorded. All items specified in 5.11.2 above shall be checked.

1) All samples which require thermal preservation shall be considered acceptable if the arrival temperature is either within +/-2°C of the required temperature or the method specified range. For samples with a specified temperature of 4°C, samples with a temperature ranging from just above the freezing temperature of water to 6°C shall be acceptable. Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun such as arrival on ice.

2) The laboratory shall implement procedures for checking chemical preservation using readily available techniques, such as pH, or free chlorine or temperature, prior to or during sample preparation or analysis.

b) The results of all checks shall be recorded.

c) Where there is any doubt as to the item's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory should consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet the sample receipt acceptance criteria listed in 5.11.3.a, 5.11.3.b or 5.11.3.c, the laboratory shall either:

1) Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or

2) Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.

i. The condition of these samples shall, at a minimum, be noted on the chain of custody or transmittal form and laboratory receipt documents.

ii. The analysis data shall be appropriately "qualified" on the final report.

d) The laboratory shall utilize a permanent chronological record such as a log book or electronic database to document receipt of all sample containers.

1) This sample receipt log shall record the following:

i. Client/Project Name

ii. Date and time of laboratory receipt

iii. Unique laboratory ID code (see 5.11.1)

iv. Signature or initials of the person making the entries.

2) During the log in process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and readily available to individuals who will process the sample. Note: the placement of the laboratory ID number on the sample container is not considered a permanent record.

i. The field ID code which identifies each container must be linked to the laboratory ID code in the sample receipt log.

ii. The date and time of sample collection must be linked to the sample container and to the date and time of receipt in the laboratory.

iii. The requested analyses (including applicable approved test method numbers) must be linked to the laboratory ID code.

iv. Any comments resulting from inspection for sample rejection shall be linked to the laboratory ID code.

e) All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.

f) A complete chain of custody record (Section 5.12.4), if utilized, shall be maintained.

5.11.4 Storage Conditions

The laboratory shall have documented procedures and appropriate facilities to avoid deterioration, contamination, or damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item shall be followed. Where items have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored and recorded where necessary.

a) Samples shall be stored according to the conditions specified by preservation protocols:

1) Samples which require thermal preservation shall be stored under refrigeration which is +/-2° of the specified preservation temperature unless method specific criteria exist. For samples with a specified storage temperature of 4°C, storage at a temperature above the freezing point of water to 6°C shall be acceptable.

2) Samples shall be stored away from all standards, reagents, food and other potentially contaminating sources. Samples shall be stored in such a manner to prevent cross contamination.

b) Sample fractions, extracts, leachates and other sample preparation products shall be stored according to 5.11.4.a above or according to specifications in the test method.

c) Where a sample or portion of the sample is to be held secure (for example, for reasons of record, safety or value, or to enable check calibrations or tests to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured items or portions concerned.

5.11.5 Sample Disposal

The laboratory shall have standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products.

5.12 RECORDS

The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records which document all laboratory activities. The laboratory shall retain on record all original observations, calculations and derived data, calibration records and a copy of the test report for ~~an appropriate period~~a minimum of 5 years.

There are two levels of record keeping: 1) sample custody or tracking and 2) legal or evidentiary chain of custody. All essential requirements for sample custody are outlined in Sections 5.12.1, 5.12.2 and 5.12.3. The basic requirements for legal chain of custody (if required or implemented) are specified in Section 5.12.4.

5.12.1 Record Keeping System and Design

The record keeping system must allow historical reconstruction of all laboratory activities that produced the resultant sample analytical data. The history of the sample must be readily understood through the documentation. This shall include interlaboratory transfers of samples and/or extracts.

a) The records shall include the identity of personnel involved in sampling, preparation, calibration or testing.

b) All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.

c) The record keeping system shall facilitate the retrieval of all working files and archived records for inspection and verification purposes.

d) All documentation entries shall be signed or initialed by responsible staff. The reason for the signature or initials shall be clearly indicated in the records such as “sampled by”, “prepared by”, or “reviewed by”).

e) All generated data except those that are generated by automated data collection systems, shall be recorded directly, promptly and legibly in permanent ink.

f) Entries in records shall not be obliterated by methods such as erasures, overwritten files or markings. All corrections to record-keeping errors shall be made by one line marked through the error. The individual making the correction shall sign (or initial) and date the correction. These criteria also shall apply to electronically maintained records.

g) Refer to 5.10.6 for Computer and Electronic Data.

5.12.2 Records Management and Storage

a) All records (including those pertaining to calibration and test equipment), certificates and reports shall be safely stored, held secure and in confidence to the client. NELAP-related records shall be available to the accrediting authority.

b) All records, including those specified in 5.12.3 and 5.12.4, shall be retained for a minimum of five years from last use. All information necessary for the historical reconstruction of data must be maintained by the laboratory. Records which are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

c) Records that are stored or generated by computers or personal computers (PCS) shall have hard copy or write-protected backup copies.

d) The laboratory shall establish a record management system for control of laboratory notebooks; instrument logbooks; standards logbooks; and records for data reduction, validation storage and reporting;

e) Access to archived information shall be documented with an access log. These records shall be protected against fire, theft, loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.

f) The laboratory shall have a plan to ensure that the records are maintained or transferred according to the clients’ instructions (see 4.1.8.e) in the event that a laboratory transfers ownership or goes out of business.

5.12.3 Laboratory Sample Tracking

5.12.3.1 Sample Handling

A record of all procedures to which a sample is subjected while in the possession of the laboratory shall be maintained. These shall include but are not limited to all records pertaining to:

a) Sample preservation including appropriateness of sample container and compliance with holding time requirement;

b) Sample identification, receipt, acceptance or rejection and log-in;

c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records;

d) Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

e) Sample analysis;

f) Standard and reagent origin, receipt, preparation, and use;

g) Equipment receipt, use, specification, operating conditions and preventative maintenance;

h) Calibration criteria, frequency and acceptance criteria;

i) Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

j) Method performance criteria including expected quality control requirements;

k) Quality control protocols and assessment;

l) Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

m) All automated sample handling systems; and

~~n) Disposal of hazardous samples including the date of sample or subsample disposal and name of the responsible person.~~

n) The laboratory shall have documented procedures for the receipt, retention or safe disposal of calibration or test items, including all provisions necessary to protect the integrity of the laboratory.

5.12.3.2 Laboratory Support Activities

In addition to documenting all the above-mentioned activities, the following shall be retained:

a) All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

b) A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

c) Copies of final reports;

d) Archived standard operating procedures;

e) Correspondence relating to laboratory activities for a specific project;

f) All corrective action reports, audits and audit responses;

g) Proficiency test results and raw data; and

h) Data review and cross checking.

5.12.3.3 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, shall include:

a) Laboratory sample ID code;

b) Date and time of analysis;

c) Instrumentation identification and instrument operating conditions/parameters (or reference to such data);

d) Analysis type;

e) All manual calculations ; and

f) Analyst's or operator's initials/signature.

5.12.3.4 Administrative Records

The following shall be maintained:

a) Personnel qualifications, experience and training records;

b) ~~Initial and continuing~~Records of demonstration of ~~proficiency~~capability for each analyst; and

c) A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

5.12.4 ~~Legal or Evidentiary~~Legal/Evidentiary Custody

The use of legal chain of custody (COC) protocols ~~is strongly recommended~~ and may be required by some state or federal programs. In addition to the records listed in 5.12.3 and the performance standards outlined in 5.12.1 and 5.12.2, the following protocols shall be incorporated if legal COC is implemented by the organization.

5.12.4.1 Basic Requirements

The legal chain of custody records shall establish an intact, continuous record of the physical possession, storage and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For ease of discussion, the above-mentioned items shall be referred to as samples:

a) A sample is in someone's custody if:

1) It is in one's actual physical possession;

2) It is in one's view, after being in one's physical possession;

3) It is in one's physical possession and then locked up so that no one can tamper with it;

4) It is kept in a secured area, restricted to authorized personnel only.

b) The COC records shall account for all time periods associated with the samples.

c) The COC records shall identify ~~all~~ individuals who physically handled individual samples.

d) In order to simplify record-keeping, the number of people who physically handle the sample should be minimized. A designated sample custodian, who is responsible for receiving, storing and distributing samples is recommended.

e) The COC records are not limited to a single form or document. However, organizations should attempt to limit the number of documents that would be required to establish COC.

f) Legal chain of custody shall begin at the point established by the federal or state oversight program. This may begin at the point that cleaned sample containers are provided by the laboratory or the time sample collection occurs.

g) The COC forms shall remain with the samples during transport or shipment.

h) If shipping containers and/or individual sample containers are submitted with sample custody seals, and any seals are not intact, the lab shall note this on the chain of custody.

i) Mailed packages should be registered with return receipt requested. If packages are sent by common carrier, receipts should be retained as part of the permanent chain-of-custody documentation.

j) Once received by the laboratory, laboratory personnel are responsible for the care and custody of the sample and must be prepared to testify that the sample was in their possession and view or secured in the laboratory at all times from the moment it was received from the custodian until the time that the analyses are completed or the sample is disposed.

5.12.4.2 Required Information in Custody Records

In addition to the information specified in 5.11.1.a and 5.11.1.b, tracking records shall include, by direct entry or linkage to other records:

a) Time of day and calendar date of each transfer or handling procedure;

b) Signatures of all personnel who physically handle the sample(s);

c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and

d) Common carrier documents.

5.12.4.3 Controlled Access to Samples

Access to all legal samples and subsamples shall be controlled and documented.

a) A clean, dry, isolated room, building, and/or refrigerated space that can be securely locked from the outside must be designated as a custody room.

b) Where possible, distribution of samples to the analyst performing the analysis must be made by the custodian(s).

c) The laboratory area must be maintained as a secured area, restricted to authorized personnel only.

d) Once the sample analyses are completed, the unused portion of the sample, together with all identifying labels, must be returned to the custodian. The returned tagged sample must be retained in the custody room until permission to destroy the sample is received by the custodian or other authority.

5.12.4.4 Transfer of Samples to Another Party

Transfer of samples, subsamples, digestates or extracts to another party are subject to all of the requirements for legal chain of custody.

5.12.4.5 Sample Disposal

a) If the sample is part of litigation, disposal of the physical sample shall occur only with the concurrence of the affected legal authority, sample data user and/or submitter of the sample.

b) All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.

c) Records shall indicate the date of disposal, the nature of disposal (such as sample depleted, sample disposed in hazardous waste facility, or sample returned to client), and the name of the individual who performed the task.

5.13 LABORATORY REPORT FORMAT AND CONTENTS

The results of each test, or series of tests carried out by the laboratory shall be reported accurately, clearly, unambiguously and objectively. The results shall normally be reported in a test report and shall include all the information necessary for the interpretation of the test results and all information required by the method used. Some regulatory reporting requirements or formats such as monthly operating reports, may not require all items listed below, however, the laboratory shall provide all the required information to their client for use in preparing such regulatory reports.

a) Except as discussed in 5.13.b), each report to an outside client shall include at least the following information (those prefaced with “where relevant” are not mandatory):

1) a title, e.g., "Test Report", or "Test Certificate", "Certificate of Results" or "Laboratory Results";

2) name and address of laboratory, and location where the test was carried out if different from the address of the laboratory and phone number with name of contact person for questions;

3) unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;

This requirement may be presented in several ways:

i. The total number of pages may be listed on the first page of the report as long as the subsequent pages are identified by the unique report identification and consecutive numbers, or

ii. Each page is identified with the unique report identification, the pages are identified as a number of the total report pages (example: 3 of 10, or 1 of 20).

Other methods of identifying the pages in the report may be acceptable as long as it is clear to the reader that discrete pages are associated with a specific report, and that the report contains a specified number of pages.

4) name and address of client, where appropriate and project name if applicable;

5) description and unambiguous identification of the tested sample including the client identification code;

6) identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature;

7) date of receipt of sample, date and time of sample collection, date(s) of performance test, and time of sample preparation and/or analysis if the required holding time for either activity is less than or equal to 48 hours;

8) identification of the test method used, or unambiguous description of any non-standard method used;

9) if the laboratory collected the sample, reference to sampling procedure;

10) any deviations from (such as failed quality control), additions to or exclusions from the test method (such as environmental conditions), and any non-standard conditions that may have affected the quality of results, and including the use and definitions of data qualifiers.

11) measurements, examinations and derived results, supported by tables, graphs, sketches and photographs as appropriate, and any failures identified; identify whether data are calculated on a dry weight or wet weight basis; identify the reporting units such as μg/l or mg/kg; and for Whole Effluent Toxicity, identify the statistical package used to provide data.

12) when required, a statement of the estimated uncertainty of the test result;

13) a signature and title, or an equivalent electronic identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;

14) at the laboratory's discretion, a statement to the effect that the results relate only to the items tested or to the sample as received by the laboratory;

15) at the laboratory's discretion, a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory;

16) clear identification of all test data provided by outside sources, such as subcontracted laboratories, clients, etc; and

17) clear identification of numerical results with values ~~below 3.18 times the MDL (10 standard deviations as determined by the method detection limit study)~~outside of quantitation levels.

b) Laboratories that are operated by a facility and whose sole function is to provide data to the facility management for compliance purposes (in-house or captive laboratories) shall have all applicable information specified in 1 through 17 above readily available for review by the accrediting authority. However formal reports detailing the information are not required if:

1) The in-house laboratory is itself responsible for preparing the regulatory reports; or

2) The laboratory provides information to another individual within the organization for preparation of regulatory reports. The facility management must ensure that the appropriate report items are in the report to the regulatory authority if such information is required.

c) Where the certificate or report contains results of tests performed by sub-contractors, these results shall be clearly identified by subcontractor name or applicable accreditation number.

d) After issuance of the report, the laboratory report shall remain unchanged. Material amendments to a calibration certificate, test report or test certificate after issue shall be made only in the form of a further document, or data transfer including the statement "Supplement to Test Report or Test Certificate, serial number . . . [or as otherwise identified]", or equivalent form of wording. Such amendments shall meet all the relevant requirements of this Standard.

e) The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.

f) The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.

g) Laboratories accredited to be in compliance with these standards shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

5.14 SUBCONTRACTING ANALYTICAL SAMPLES

a) The laboratory shall advise the client in writing of its intention to sub-contract any portion of the testing to another party.

b) Where a laboratory sub-contracts any part of the testing covered under NELAP, this work shall be placed with a laboratory accredited under NELAP for the tests to be performed.

c) The laboratory shall retain records demonstrating that the above requirements have been met.

5.15 OUTSIDE SUPPORT SERVICES AND SUPPLIES

a) Where the laboratory procures outside services and supplies, other than those referred to in this Standard, in support of tests, the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's tests.

b) Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials and services comply with specified requirements. The laboratory should, wherever possible, ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated or otherwise verified as complying with any standard specifications relevant to the calibrations or tests concerned.

c) The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for tests.

5.16 COMPLAINTS

The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this Standard or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with Section 5.5.3.1. Records of the complaint and subsequent actions shall be maintained.

QUALITY SYSTEMS
APPENDIX A

REFERENCES

Appendix A - REFERENCES

40 CFR Part 136, Appendix A, paragraphs 8.1.1 and 8.2

American Association for Laboratory Accreditation April 1996. General Requirements for Accreditation

“American National Standards Specification and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs (ANSI/ASQC E-4)”, 1994

Catalog of Bacteria, American Type Culture Collection, Rockville, MD

EPA 2185 - Good Automated Laboratory Practices, 1995 available at www.epa.gov/docs/etsdwe1/irm_galp/

“Glossary of Quality Assurance Terms and Acronyms”, Quality Assurance Division, Office of Research and Development, USEPA

"Guidance on the Evaluation of Safe Drinking Water Act Compliance Monitoring Results from Performance Based Methods", September 30, 1994, Second draft.

International vocabulary of basic and general terms in metrology (VIM): 1984. Issued by BIPM. IEC. ISO. and OIML

ISO Guide 3534-1: “Statistics, vocabulary and symbols - Part 1: Probability and general statistical terms”

ISO Guide 7218: Microbiology - General Guidance for Microbiological Examinations

ISO Guide 8402: 1986. Quality - Vocabulary

ISO Guide 9000: 1994 Quality management and quality assurance standards - Guidelines for selection and use

ISO Guide 9001: 1994 Quality Systems - Model for quality assurance in design/development, production, installation and servicing

ISO Guide 9002: 1994 Quality systems - Model for quality assurance in production and installation

ISO/IEC Guide 2: 1986. General terms and their definitions concerning standardization and related activities

ISO/IEC Guide 25: 1990. General requirements for the competence of calibration and testing laboratories

“Laboratory Biosafety Manual”, World Health Organization, Geneva, 1983

Manual for the Certification of Laboratories Analyzing Drinking Water Revision 4, EPA 815-B-97-001

Manual of Method for General Bacteriology, Philipp Gerhard et al., American Society for Microbiology, Washington, 1981

Performance Based Measurement System, EPA EMMC Method Panel, PBM workgroup, 1996

QUALITY SYSTEMS
APPENDIX B

DEFINITIONS FOR QUALITY SYSTEMS

Appendix B - DEFINITIONS FOR QUALITY SYSTEMS

The following definitions are used in the text of Quality Systems. In writing this document, the following hierarchy of definition references were used: ISO 8402, ANSI/ASQC E-4, EPA's Quality Assurance Division Glossary of Terms, and finally definitions developed by NELAC ~~and/or the Quality Assurance Standing Committee~~. The source of each definition, unless otherwise identified, is ~~noted~~the Quality Systems Committee.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accrediting Authority: the agency having responsibility and accountability for environmental laboratory accreditation and who grants accreditation. For the purposes of NELAC, this is EPA, other federal agencies, or the state. (NELAC)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes ~~a~~ combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Analysis Duplicate: the second measurement of the target analyte(s) performed on a single sample or sample preparation.

Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (Applicable only to radiochemistry)

Analytical Reagent (AR) Grade: designation for the high purity of certain chemical reagents and solvents given the American Chemical Society. ~~(Quality Systems)~~

Assessor Body: the organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency testing results, surveys the site, etc., whether EPA, the state, or contracted private party. (NELAP)

Batch: environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include ~~prepared~~ samples originating from various environmental matrices and can exceed 20 samples. ~~(Quality Systems)~~

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Blind Sample: a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibrate: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration: the set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurand. (VIM - 6.13)

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response.

Calibration Standard: a ~~solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The Calibration solutions are~~substance or reference material used to calibrate ~~the~~an instrument ~~response with respect to analyte concentration~~. (Glossary of Quality Assurance Terms and Acronyms, ~~QAMS, 8/31/92~~12/6/95).

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

~~**Chain of Custody:** an unbroken trail of accountability that documents the physical security of samples, data and records.~~

Confirmation: verification of the ~~presence~~presence/identity of a component ~~through the use of an analytical technique that differs from the original test method. These~~approach that may include:

- Second column confirmation
- Alternate wavelength
- Derivatization
- Mass spectral interpretation
- Alternative detectors or
- Additional cleanup procedures~~.~~.
- Alternative technique or conditions.

Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.)

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form.

Desorption Efficiency: The mass of target analyte recovered from sampling media, usually a sorbent tube, divided by the mass of target analyte spiked on to the sampling media expressed as a percentage. Sample target analyte masses are usually adjusted for the desorption efficiency.

Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. ~~See Method Detection Limit.~~

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Environmental Detection Limit (EDL): the smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (Radioanalysis Subcommittee)

Field Duplicate: A second sample collected simultaneously with the original sample (air testing) or immediately after the original sample (other matrices) from the same location as the original sample. The results of field duplicate analyses are used to evaluate the repeatability of the test method and to provide information about the homogeneity of the site samples.

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136).

~~Initial~~ **Demonstration of Capability**: procedure to establish the ability of the laboratory to generate data of acceptable accuracy and precision ~~which is included in many of the EPA's analytical test methods. In general the procedure includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and the percentage recovery and the standard deviation are determined and compared to specified limits. (40 CFR Part 136)~~.

Internal Standard: a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method.

Laboratory: Body that calibrates and/or tests**.**

NOTES:
1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.
2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing
- at or from a permanent location,
- at or from a temporary facility, or
- in or from a mobile facility. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank or spiked blank): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (NELAC).

Laboratory Duplicate: Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records. For the purpose of litigation, this includes the signatures of all who handle the samples (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Limit of Detection (LOD): the lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also ~~Method~~ Detection Limit (D.1.4).

Manager (however named): the individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.

Matrix: The component or substrate ~~which~~that may contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:
- Aqueous: Any aqueous sample excluded from the definition of a drinking water matrix or Saline/Estuarine source. Includes surface water, groundwater and effluents.
- Drinking water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15% settleable solids.
- Chemical Waste: A product or by-product of a industrial process that results in a matrix not previously defined.
- Air ~~Samples: Media used to retain the analyte~~: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from ~~an air sample such as sorbent tubes or summa canisters. Each medium~~

~~shall be considered as a distinct matrix. (Quality Systems)~~a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device.

Matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

May: permitted, but not required (TRADE)

Media: material that supports the growth of a microbiological culture.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) ~~that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of~~in which no target analytes or interferences are presnt at concentrations that impact the analytical ~~procedures.~~ results for sample analyses (NELAC).

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B).

Must: denotes a requirement that must be met. (Random House College Dictionary)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

NELAP: the overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

Performance Audit: the routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.

Positive Control: measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC).

Preservation: ~~refrigeration and or reagents added at the time of sample collection~~sample handling and/or treatment such as temperature control, addtion of reagents, packaging and protection from light, needed to maintain the chemical and or biological integrity of the sample.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria . (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Proficiency Testing: Determination of the laboratory calibration or testing performance by means of interlaboratory comparisons. (ISO/IEC Guide 2 - 12.6, amended)

Proficiency Testing Program: the aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.

Protocol: a detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Pure Reagent Water: shall be water in which no target analytes or interferences are present at a concentration which would impact the results when using a particular analytical test method.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Quality Manual: A document stating the quality policy, quality system and quality practices of an organization. This may be also called a Quality Assurance Plan or a Quality Plan.

> NOTE - The quality manual may call up other documentation relating to the laboratory's quality arrangements.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target ~~variable (e.g., target~~ analyte~~)~~ that can be quantified with the ~~confidence level~~accuracy required by the data ~~user. Quantitation limit, for the purposes of NELAC, is defined as 3.18 times the MDL, by convention~~

Quality Control Sample: an uncontaminated sample with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Range: the difference between the minimum and the maximum of a set of values.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes ,or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30 - 2.1)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM - 6.08)

Requirement: a translation of the needs into a set of individual quantified or descriptive specifications for the characteristics of an entity in order to enable its realization and examination.

Reference Toxicant: see D.2.1.a

Sampling Media: material used to collect and concentrate the target analytes(s) during air sampling such as solid sorbents, filters, or impinger solutions.

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of nontarget substances.

Sensitivity: the capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991).

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991).

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Spike: a known mass of target analyte added to a blank, sample or subsample; used to determine recovery efficiency or for other quality control purposes.

Standard Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical test method.

Supervisor (however named): the individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

Technical Director: ~~*Definition needs to be developed*~~ (however named) has overall responsibility for the technical operation of the environmental testing laboratory.

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure.

> NOTE - The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

Test Method: defined technical procedure for performing a test.

Testing Laboratory: laboratory that performs tests. (ISO/IEC Guide 2 - 12.4)

Test Sensitivity/Power: D.2.4.a

Tolerance Chart: A chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/- 3 sigma). (ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for Radioassay Laboratories)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM - 6.12)

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE - ~~In connection with the management of measuring equipment, v~~Verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

~~**Validation: the process of substantiating specified performance criteria.**~~

Work Cell: a group of well defined analyts that together perform the method analysis. The members of the group and their specific function/s within the workcell must be fully documented.

QUALITY SYSTEMS
APPENDIX C

~~INITIAL~~ DEMONSTRATION OF CAPABILITY

Appendix C - ~~INITIAL~~ DEMONSTRATION OF CAPABILITY

C.1 PROCEDURE FOR ~~INITIAL~~ DEMONSTRATION OF CAPABILITY

~~An initial~~A demonstration of ~~method performance~~capability (DOC) must be made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method (see 5.10.2.1).

~~All initial~~Note: In laboratories with specialized "work cells" (a group of well defined analyts that together perform the method analysis), the group as a unit must meet the above criteria and this demonstration must be fully documented.

In general, this demonstration does not test the performance of the method in real world samples, but in the applicable and available clean matrix (a sample of a matrix in which no target analytes or interferences are present at concentrations that impact the results of a specific test method), e.g., water, solids, biological tissue and air. However, actual sample spike results may be used to meet this standard, i.e.,at least 4 consecutive matrix spikes within the last 12 months. In addition, for analytes which do not lead themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples.

All demonstrations, ~~continuing demonstrations and method certification~~ shall be documented through the use of the forms in this appendix.

The following steps, which are adapted from the EPA test methods published in 40 CFR Part 136, Appendix A, shall be performed if required by mandatory test method or regulation.: (Note for analytes for which spiking is not an option and for which quality control samples are not readily available, the 40 CFR approach is one way to perform this demonstration and it is the responsibility of the laboratory to document that other approaches to DOC are adequate and this shall be documented in the laboratory's Quality Manual.

a) A quality control sample shall be obtained from an outside source. If not available, the QC ~~check~~ sample may be prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The ~~concentrate~~analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the ~~required method volume~~concentration specified or if unspecified to a concentration approximately 10 times the method-stated or laboratory-calculated method detection limit.

c) ~~The~~At least four aliquots shall be prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the ~~four~~ results, calculate the ~~average~~mean recovery ($\bar{x}$) in the appropriate reporting units (such as μg/L) and the standard deviations of the population sample (n-1) (in the same units) for each parameter of interest.

~~e) For each parameter, compare s and $\bar{x}$~~ When it is not possible to determine mean and standard deviations, such as for presence absence and logarithmic values the laboratory will assess performance against established and documented criteria.

e) Compare the information from (e) above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated

acceptance criteria (if there a~~ non-standard method~~re not established mandatory criteria). If~~ s and x for~~ all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters ~~exceed~~do not meet the acceptance ~~range~~criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to 1) or 2) below.

1) Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.

2) Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

C.2 CERTIFICATION STATEMENT

The following certification statement shall be used to document the completion of each ~~initial~~ demonstration of capability. A copy of the certification statement shall be retained in the personnel records of each affected employee (see 5.6.3 and 5.12.3.4.b).

Initial Demonstration of Capability
Certification Statement

Date: Page __of __
Laboratory Name:
Laboratory Address:
Analyst(s) Name(s):

Matrix:
(examples: laboratory pure water, soil, air, ~~waste~~ solid, ~~leachate, sludge, other~~biological tissue)
Method number, and Analyte, or Class of Analytes or Measured Parameters
(examples: barium by 200.7, trace metals by 6010, benzene by 8021, etc.)

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method/s, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the ~~Initial~~ Demonstration of Capability.

2. The test method/s was performed by the analyst(s) identified on this certification.

3. A copy of the test method/s and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the ~~initial~~ demonstration capability are true, accurate, complete and self-explanatory (1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

_____	_____	_____
Technical Director's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time ~~an initial~~a demonstration of capability study is completed.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

QUALITY SYSTEMS
APPENDIX D

ESSENTIAL QUALITY CONTROL REQUIREMENTS

Appendix D - ESSENTIAL QUALITY CONTROL REQUIREMENTS

The quality control protocols specified by the laboratory's method manual (5.10.1.2) shall be followed. The laboratory shall ensure that the essential standards outlined in Appendix D are incorporated into their method manuals

All quality control measures shall be assessed and evaluated on an on-going basis and quality control acceptance criteria shall be used to determine the validity of the data. The laboratory shall have procedures for the development of acceptance/rejection criteria where no method or regulatory criteria exists.

The requirements from the body of Chapter 5, e.g., 5.5.4, apply to all types of testing. The specific manner in which they are implemented is detailed in each of the sections of this Appendix, i.e., chemical testing, W.E.T. testing, microbiology testing, radiochemical testing and air testing.

D.1 CHEMICAL TESTING

D.1.1 Positive and Negative Controls

a) Negative Controls

1) Method Blanks - Shall be performed at a frequency of one per batch of samples per matrix type per sample extraction or preparation method. The results of this analysis shall be one of the QC measures to be used to assess batch acceptance. The source of contamination must be investigated and measures taken to correct, minimize or eliminate the problem if

i) the blank contamination exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated sample batch ~~and~~or

ii) the blank contamination exceeds the concentration present in the samples and is greater than 1/10 of the specified regulatory limit.

Each sample in the affected batch must be assessed against the above criteria to determine if the sample datum is acceptable. Any sample associated with the contaminated blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

b) Positive Controls

1) Laboratory Control Sample - (QC Check Samples) Shall be analyzed at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance. NOTE: the Matrix spike (see 2 below) may be used ~~as a~~in place of this control as long as the acceptance criteria are as stringent as for the LCS.

2) Matrix Spikes (MS) - Shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for

which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike.

3) Surrogates - Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with the sample composition and shall be reported to the client whose sample produced the poor recovery.

4) If the mandated or requested test method does not specify the spiking ~~compounds~~components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses ~~and shall include~~, permit specified analytes and other client requested components. ~~T~~However, the laboratory shall ensure~~, however,~~ that all reported components are used in the spike mixture within a two-year time period~~, and that no one component or components dominate the spike mixture~~.

D.1.2 Analytical Variability/Reproducibility

Matrix Spike Duplicates (MSDs) or Laboratory Duplicates - Shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate.

D.1.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) ~~Initial~~ Demonstration of Analytical Capability - (Section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel, matrix or test method.

b) Calibration - Calibration protocols specified in Section 5.9.4 shall be followed.

c) Proficiency Test Samples - The results of such analyses (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.1.4 ~~Method~~ Detection Limits

~~Method~~The laboratory shall utilize a test method that provides a detection ~~limits (MDL)~~limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by ~~40 CFR Part 136, Appendix B unless included~~the protocol in ~~a~~the mandated test method or ~~program.~~

~~a) applicable regulation, e.g., MDL. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.~~

a) An MDL study is not required for any component for which spiking solutions or quality control samples are not available such as ~~total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature dissolved oxygen or turbidity.~~

~~b) The method~~odor and temperature.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a ~~clean matrix appropriate to the test method (such as laboratory pure reagent water or Ottawa sand) or~~matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

~~c) All quantitatively reported results (i.e., those greater than 3.18 times the MDL) shall be bracketed by calibration or calibration verification standards.~~

~~d) The MDL shall be verified annually by the preparation and analysis of at least one clean matrix sample spiked at the current reported MDL. If the selected components cannot be detected, the MDL study must be repeated.~~

~~e~~c) The test method's quantitation limits (see 5.9.4.2.1.f) must be established and must be above the established detection limit.

d) Detection limits must be determined each time there is a significant change in the test method or instrument type.

e) It is essential that all sample processing steps of the analytical method be included in the determination of the detection limit.

f) All procedures used must be documented ~~including~~. Documentation must include the matrix type. All supporting data must be retained.

g) The laboratory must have established procedures to tie detection limits with quantitation limits.

D.1.5 Data Reduction

The procedures for data reduction, such as use of linear regression, shall be documented.

D.1.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.9.2.

b) Reagent Quality, Water Quality and Checks:

1) Reagents - In methods where the purity of reagents is not specified, analytical reagent grade shall be used. Reagents of lesser purity than those specified by the test method shall not be used. The labels on the container should be checked to verify that the purity of the reagents meets the requirements of the particular test method. Such information shall be documented.

2) Water - The quality of water sources shall be monitored and documented and shall meet method specified requirements.

D.1.7 Selectivity

a) Absolute retention time and relative retention time aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column to separate constituents. The laboratory shall develop and document acceptance criteria for retention time windows.

b) A confirmation shall be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested by the laboratory. Such confirmations shall be performed on organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. Confirmation is required unless stipulated in writing by the client. All confirmation shall be documented.

c) The laboratory shall ~~develop and~~ document acceptance criteria for mass spectral tuning.

D.1.8 Constant and Consistent Test Conditions

a) The laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used .

b) Glassware Cleaning - Glassware shall be cleaned to meet the sensitivity of the test method.

Any cleaning and storage procedures that are not specified by the test method shall be documented in laboratory records and SOPs.

D.2 WHOLE EFFLUENT TOXICITY

D.2.1 Positive and Negative Controls

a) Positive Control - Reference Toxicants - Reference toxicant tests indicate the sensitivity of the test organisms being used and demonstrate a laboratory's ability to obtain consistent results with the test method.

1) The laboratory must demonstrate its ability to obtain consistent results with reference toxicants before it performs toxicity tests with effluents for permit compliance purposes.

i. An intralaboratory coefficient of variation (%CV) is not established for each test method. However, a testing laboratory shall maintain control charts for the control performance and reference toxicant statistical endpoint (such as NOEC or ECp) and shall evaluate the intralaboratory variability with a specific reference toxicant for each test method. In addition, a laboratory must produce test results that meet test acceptability criteria (such as greater than 80% survival in the control) as specified in the specific test method.

ii. Intra-laboratory precision on an ongoing basis must be determined through the use of reference toxicant tests and plotted in quality control charts. As specified in the test methods, the control charts shall be plotted as point estimate values, such as EC25 for chronic tests and LC 50 for acute tests, over time within a laboratory.

2) The frequency of reference toxicant testing shall comply with the EPA or state permitting authority requirements.

3) The USEPA test methods for EPA/600/4-91-002, EPA/600/4-91-003 and EPA/600/4-90-027F do not currently specify a particular reference toxicant and dilution series, however, if the state or permitting authority identifies a reference toxicant or dilution series for a particular test, the laboratory shall follow the specified requirements.

4) Test Acceptability Criteria (TAC) - The test acceptability criteria (for example, the chronic *Ceriodaphnia* test, requires 80% or greater survival and an average 15 young per female in the controls) as specified in the test method must be achieved for both the reference toxicant and effluent test. The criteria shall be calculated and shall meet the method specified requirements for performing toxicity:

i. The control population of *Ceriodaphnia* shall contain no more than 20% males.

ii. An individual test may be conditionally acceptable if temperature, dissolved oxygen, pH and other specified conditions fall outside specifications, depending on the degree of the departure and the objectives of the tests (see test conditions and test acceptability criteria specified for each test method). The acceptability of the test shall depend on the experience and professional judgment of the technical employee and the permitting authority.

b) Negative Control - Control, Brine Control or Dilution Water - The standards for the use, type and frequency of testing are specified by the test methods and by permit and shall be followed.

D.2.2 Variability and/or Reproducibility

Intra-laboratory precision shall be determined on an ongoing basis through the use of further reference toxicant tests and related control charts as described in item D.2.1.a above.

D.2.3 Accuracy

This principle is not applicable to Whole Effluent Toxicity.

D.2.4 Test Sensitivity

a) Test sensitivity (or test power) of the tests will depend in part on the number of replicates per concentration, the significance level selected (0.05), and the type of statistical analysis. If the variability remains constant, the sensitivity of the test will increase as the number of replicates is increased. Test sensitivity is the minimum significant difference (MSD) between the control and test concentration that is statistically significant. If the Dunnett's procedure is used, the MSD shall be calculated according to the formula specified by the EPA test method and reported with the test results.

b) Estimate the MSD for non-normal distribution and or heterogenous variances.

c) Point estimates: (LCp, ICp, or ECp) - Confidence intervals shall be reported as a measure of the precision around the point estimate value.

d) The MSD shall be calculated and reported for only chronic endpoints. In addition, the calculated endpoint is typically a lethal concentration of 50% (LC 50), therefore, confidence intervals shall be reported as a measure of the precision around the point estimate value. In order to have sufficient replicates to perform a reliable MSD, such tests shall have a minimum of four replicates per treatment so that either parametric or non parametric tests can be conducted.

D.2.5 Selection of Appropriate Statistical Analysis Methods

a) The methods of data analysis and endpoints will be specified by language in the permit or, if not present in the permit, by the EPA methods manuals for Whole Effluent Toxicity.

b) Dose Response Curves - When required, the data shall be plotted in the form of a curve relating the dose of the chemical to cumulative percentage of test organisms demonstrating a response such as death.

D.2.6 Selection and Use of Reagents and Standards

a) The grade of all reagents used in Whole Effluent Toxicity tests is specified in the test method except the reference standard. All reference standards shall be prepared from chemicals which are analytical reagent grade or better. The preparation of all standards and reference toxicants shall be documented.

b) All standards and reagents associated with chemical measurements, such as dissolved oxygen, pH or specific conductance, shall comply with the standards outlined in Appendix D.1 above.

D.2.7 Selectivity

This principle is not applicable. The selectivity of the test is specified by permit.

D.2.8 Constant and Consistent Test Conditions

a) If closed refrigerator-sized incubators are used, culturing and testing of organisms shall be separated to avoid loss of cultures due to cross-contamination.

b) The laboratory or a contracted outside expert shall positively identify test organisms to species on an annual basis. The taxonomic reference (citation and page(s))and the names(s) of the taxonomic expert(s) must be kept on file at the laboratory.

c) Instruments used for routine measurements of chemical and physical parameters such as pH, DO, conductivity, salinity, alkalinity, hardness, chlorine, and weight shall be calibrated, and/or standardized per manufacturer's instructions and Section D.1. Temperature shall be calibrated per section 5.9.4.2.1 All measurements and calibrations shall be documented.

d) Test temperature shall be maintained as specified in the methods manuals. The average daily temperature of the test solutions must be maintained within 1°C of the selected test temperature, for the duration of the test. The minimum frequency of measurement shall be once per 24 hour period. The test temperature for continuous flow toxicity tests shall be recorded and monitored continuously.

e) Water used for culturing and testing shall be analyzed for toxic metals and organics annually or whenever the minimum acceptability criteria for control survival, growth or reproduction are not met and no other cause, such as contaminated glassware or poor stock, can be identified. The method specified analytes and concentration levels shall be followed.

f) New batches of food used for culturing and testing shall be analyzed for toxic organics and metals. If food combinations or recipes are used, analyses shall be performed on the final product upon the use of new lot of any ingredient. If the concentration of total organic chlorine exceeds 0.15 μg/g wet weight, or the total concentration of organochlorine pesticides plus PCBs exceeds 0.30 μg/g wet weight, or toxic metals exceeds 20 μg/g wet weight, the food must not be used.

g) Test chamber size and test solution volume shall be as specified in the methods manuals.

h) Test organisms shall be fed the quantity and type food specified in the methods manuals. They shall also be fed at the intervals specified in the test methods.

i) Light intensity shall be maintained as specified in the methods manuals. Measurements shall be made and recorded on a yearly basis. Photoperiod shall be maintained as specified in the test methods and shall be documented at least quarterly. For algal tests, the light intensity shall be measured and recorded at the start of each test.

j) At a minimum, during chronic testing DO and pH shall be measured daily in at least one replicate of each concentration. DO may be measured in new solutions prior to organism transfer, in old solutions after organisms transfer, or both.

k) All cultures used for testing shall be maintained as specified in the methods manuals.

l) Age and the age range of the test organisms must be as specified in the manuals.

m) The maximum holding time (lapsed time from sample collection to first use in a test) shall not exceed 36 hours without the permission of the permitting authority.

n) All samples shall be chilled to 4°C during or immediately after collection. They shall be maintained at a temperature range from just above the freezing temperature of water to 6°C and the arrival temperature shall be no greater than 6°C. Samples that are hand delivered to the laboratory immediately after collection (i.e., within 1 hour) may not meet the laboratory temperature acceptance criteria. In these cases, the laboratory may accept the samples if there is evidence (such as arrival on ice) that the chilling process has begun.

o) Organisms obtained from an outside source must be from the same batch.

D.3 MICROBIOLOGY

These standards apply to laboratories undertaking the examination of materials, products and substances involving microbiological analysis, recovery or testing. The procedures involve the culture media, the test sample and the microbial species being isolated, tested or enumerated.

a) Microbiological testing refers to and includes the detection, isolation, enumeration and identification of microorganisms and their metabolites, as well as sterility testing. It includes assays using microorganisms as part of a detection system and their use for ecological testing.

b) These standards are concerned with the quality of test results and not specifically with health and safety measures. In the performance of microbiological testing, laboratories must be aware of and have SOPs that conform with local, state, and national regulatory policies for the safety and health of personnel.

~~c) Clothing appropriate to the type of testing being performed shall be worn, and often includes protection for hair, beard, hands and shoes. Protective clothing worn in the microbiological laboratory shall be removed before leaving the restricted area.~~

D.3.1 Positive and Negative Controls

a) Negative Controls

The laboratory shall demonstrate that the cultured samples have not been contaminated through ~~sampling~~sample handling/preparation or environmental exposure. These controls shall include sterility checks of media ~~and~~, blanks such as filtration blanks, bottle, and buffer blanks.

1) All blanks and uninoculated controls specified by the test method shall be prepared and analyzed at the frequency stated in the method.

2) A minimum of one uninoculated control shall be prepared and analyzed unless the same equipment set is used to prepare multiple samples. In such cases, the laboratory shall prepare a series of blanks using the equipment. At least one beginning and ending control shall be prepared, with additional controls inserted after every 10 samples.

3) Analyze a known negative culture.

b) Positive Controls

Positive controls demonstrate that the medium can support the growth of the test organism, and that the medium produces the specified or expected reaction to the test organism.

1) On a monthly basis each lot of media shall be tested with at least one pure culture of a known positive reaction and shall be included with the sample test batch.

2) If routine culturing is not part of a laboratory's testing and pre-prepared media are routinely used, strict control of the storage conditions and expiration date of media shall be maintained. A positive growth control from a know positive sample shall be run with each test batch to ensure that the media support growth.

3) If the laboratory has at least one known positive result of the appropriate organism during the month, a separate positive control is not required.

D.3.2 Test Variability/Reproducibility

a) Duplicates - At least 5% of the suspected positive samples shall be duplicated. In laboratories with more than one analyst, each shall make parallel analyses on at least one positive sample per month.

b) Where possible, participation in, or organization of collaborative trails, proficiency testing, or interlaboratory comparisons, either formal or informal, must be done.

D.3.3 Method Evaluation

a) In order to demonstrate the suitability of a test method for its intended purpose, the laboratory shall demonstrate and document its ability to meet acceptance criteria either specified by the method or by the EPA or State program requirements. Acceptance criteria must meet or exceed these requirements and must demonstrate that the test method provides correct/expected results with respect to specified detection capabilities, selectivity, and reproducibility.

1) Accepted (official) test methods or commercialized test kits for official test methods, or test methods from recognized national or international standard organizations, may not require a specific validation. Laboratories are required, however, to demonstrate proficiency with the test method prior to first use. This can be achieved by simultaneous, side-by-side analysis by several analysts.

2) Qualitative microbiological test methods in which the response is expressed in terms of presence/absence, shall be validated by estimating, if possible, the specificity, and reproducibility. The differences due to the matrices must be taken into account when testing different sample types.

3) The validation of microbiological test methods shall be performed under the same conditions as those for routine sample analysis. This can be achieved by using a combination of naturally contaminated products and spiked products with results that can be statistically analyzed to demonstrate that the test meets its intended purpose.

4) All validation data shall be recorded and stored at least as long as the test method is in force, or if withdrawn from active use, for at least 5 years past the date of last use.

b) Laboratories shall participate in the Proficiency Test programs (interlaboratory) identified by NELAP (5.4.2.j or 5.5.3.4).

D.3.4 Test Performance

All growth and recovery media must be checked to assure that the target organisms respond in an acceptable and predictable manner (see D.3.1.b).

D.3.5 Data Reduction

a) The calculations, data reduction and statistical interpretations specified by each test method shall be followed.

b) If the test method specifies colony counts, such as membrane filter or colony counting, then the ability of individual analysts to count colonies shall be verified at least once per month, by having two or more analysts count colonies from the same plate.

D.3.6 Quality of Standards, Reagents and Media

The laboratory shall ensure that the quality of the reagents and media used is appropriate for the test concerned.

a) Culture media may be prepared in the laboratory from the different chemical ingredients, from commercial dehydrated powders or may be purchased ready to use.

b) Reagents, commercial dehydrated powders and media shall be used within the shelf-life of the product and shall be documented according to 5.10.5. The laboratory shall retain all manufacturer supplied “quality specification statements” which may contain such information as shelf life of the product, storage conditions, sampling regimen/rate, sterility check including acceptability criteria, performance checks including the organism used, their culture collection reference and acceptability criteria, date of issue of specification, or statements assuring that the relevant product batch meets the product specifications.

c) Distilled water, deionized water or reverse osmosis produced water free from bactericidal and inhibitory substances shall be used in the preparation of media solutions and buffers. Where required by the test method, the quality of the water ~~(~~shall be monitored for attributes such as pH, chlorine residual, specific conductance or metals~~) shall be monitored~~ at the specified frequency and evaluated according to the stated standards. Records shall be maintained on all activities.

d) Media, solutions and reagents shall be prepared, used and stored according to a documented procedure following the manufacturer’s instructions or the test method.

e) All laboratory media shall be checked to ensure they support the growth of specific microbial cultures. In addition, selective media shall be checked to ensure they suppress the growth of non-target organisms. Media purchased pre-prepared from the manufacturer shall be checked monthly except when the use and maintenance of pure cultures is not part of laboratory procedures. In preference to using the commonly used streak method, it is

better to use a quantitative procedure, where a known (often low) number of relevant organisms are inoculated into the medium under test and the recovery evaluated.

f) Each lot of laboratory detergent shall be checked to ensure that residues from the detergent do not inhibit or promote growth of microorganisms, ~~such~~for example with a~~s~~n inhibitory residue test.

D.3.7 Selectivity

a) All confirmation/verification tests specified by the test method shall be performed according to method protocols.

b) In order to demonstrate traceability and selectivity, laboratories shall use reference cultures of microorganisms obtained from a recognized national collection or an organization recognized by the assessor body.

1) Reference cultures may be subcultured once to provide reference stocks. Appropriate purity and biochemical checks shall be made and documented. The reference stocks shall be preserved by a technique which maintains the desired characteristics of the strains. Examples of such methods are freeze-drying, liquid nitrogen storage and deep-freezing methods. Reference stocks shall be used to prepare working stocks for routine work. If reference stocks have been thawed, they must not be re-frozen and re-used.

2) Bacterial working stocks shall not be sub-cultured under normal conditions. However working stocks ~~may~~shall not be ~~subcultured up to a defined number of subcultures~~sequentially cultured more than five times except when:

i. it is required by standard test methods, or

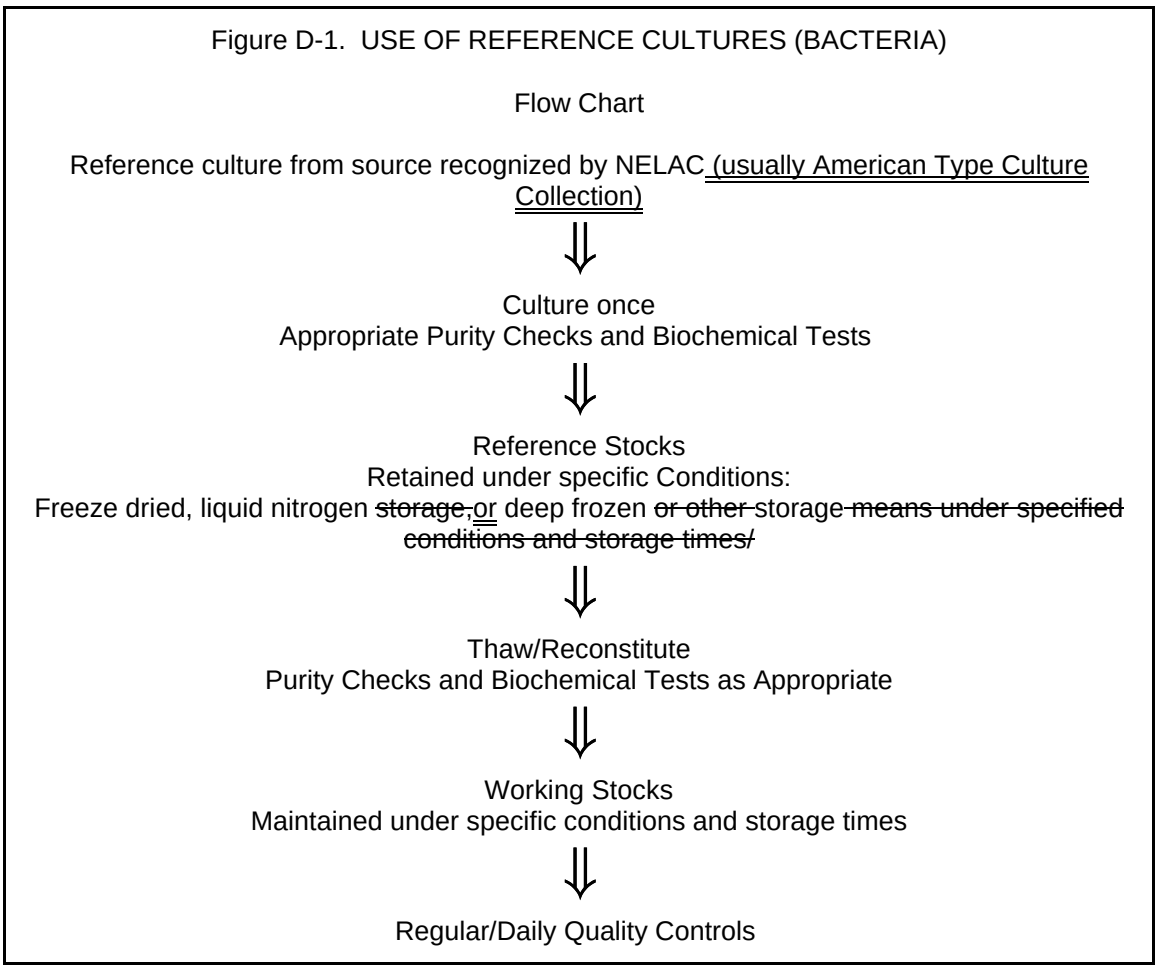
ii. laboratories can provide documentary evidence demonstrating that there has been no loss of viability, no changes in biochemical activity and/or no change in morphology.

3) Working stocks shall not be subcultured to replace reference stocks.

4) A scheme for handling reference cultures is included in figure D.1.

Figure D-1. USE OF REFERENCE CULTURES (BACTERIA)

Flow Chart



D.3.8 Constant and Consistent Test Conditions

a) The laboratory shall devise an appropriate environmental monitoring program to indicate trends in levels of contamination appropriate to the type of testing being carried out. Acceptable background counts shall be determined and there shall be a documented procedures to deal with situations in which these limits are exceeded.

b) Walls, floors, ceilings and work surfaces shall be non-absorbent and easy to clean and disinfect. Wooden surfaces of fixtures and fitting shall be adequately sealed. Measures shall be taken to avoid accumulation of dust by the provision of sufficient storage space by having minimal paperwork in the laboratory and by prohibiting plants and personal possessions from the laboratory work area.

c) Temperature measurement devices

1) Where the accuracy of temperature measurement has a direct effect on the result of the analysis, temperature measuring devices such as liquid-in-glass thermometers, thermocouple, platinum resistance thermometers used in incubators, autoclaves and other equipment shall be the appropriate quality to

achieve the specification in the test method. The graduation of the temperature measuring devices must be appropriate for the required accuracy of measurement and they shall be calibrated to national or international standards for temperature (see 5.9.2.1). Calibration shall be done at least annually.

2) The stability of temperature, uniformity of temperature distribution and time required to achieve equilibrium conditions in incubators, waterbaths, ovens and temperature controlled rooms shall be established, for example, position, space between and height of stacks of Petri dishes.

d) Autoclaves

1) The performance of each autoclave shall be initially evaluated by establishing its functional properties, for example heat distribution characteristics with respect to typical uses. Autoclaves shall be capable of meeting specified temperature tolerances. Pressure cookers fitted only with a pressure gauge are not recommended for sterilization of media or decontamination of wastes.

2) Records of autoclave operations including temperature and time shall be maintained. This shall be done for every cycle. Acceptance/rejection criteria shall be established and used to evaluate the autoclave efficiency and effectiveness.

e) Volumetric equipment such as automatic dispensers, dispenser/diluters, mechanical hand pipettes and disposal pipettes may all be used in the microbiology laboratory. Regular checks as outlined in Section 5.9.4.2.1 shall be performed and documented.

f) UV Sterilizers

1) Are to be tested quarterly for effectiveness with positives (either reference cultures or positive monitoring samples) and this is to include testing of the power output of the UV bulb.

~~f~~g) Conductivity meters, oxygen meters, pH meters, hygrometers, and other similar measurement instruments shall be calibrated according to the method specified requirements (see Appendix D.1). Mechanical timers shall be checked regularly against electronic timing devices to ensure accuracy.

D.4 RADIOCHEMICAL ANALYSIS

These standards apply to laboratories undertaking the examination of environmental samples by radiochemical analysis. These procedures for radiochemical analysis may involve some form of chemical separation followed by detection of the radioactive decay of analyte (or indicative daughters) and tracer isotopes where used. For the purpose of these standards procedures for the determination of radioactive isotopes by mass spectrometry (e.g. ICP-MS or TIMS) or optical (e.g. KPA) techniques are not addressed herein.

D.4.1 Negative Controls

a) Method Blank - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The method blank result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When

the specified method blank acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~b~~a)19 and 20] will be followed. The occurrence of a failed method blank acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

b) In the case of gamma spectrometry where the sample matrix is simply aliquoted into a calibrated counting geometry the method blank shall be of similar counting geometry that is empty or filled to similar volume with ASTM Type II water to partially simulate gamma attenuation due to a sample matrix.

c) There shall be no subtraction of the required method blank [see D.4.1.a)] result from the sample results in the associated preparation or analytical batch. This does not preclude the application of any correction factor (e.g. instrument background, analyte presence in tracer, reagent impurities, peak overlap, calibration blank, etc.) to all analyzed samples, both program/project submitted and internal quality control samples. However, these correction factors shall not depend on the required method blank result in the associated analytical batch.

d) The method blank acceptance criteria [see 5.10.1.2.b)18] shall address the presumed aliquot size on which the method blank result is calculated and the manner in which the method blank result is compared to sample results of differing aliquot size.

D.4.2 Positive Controls

a) Laboratory Control Samples - Shall be performed at a frequency of one per preparation batch. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The laboratory control sample result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified laboratory control sample acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~b~~a)19 and 20] will be followed. The occurrence of a failed laboratory control sample acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

b) Matrix Spike - Shall be performed at a frequency of one per preparation batch for those methods which do not utilize an internal standard or carrier and for which there is a physical or chemical separation process and where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The matrix spike result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified matrix spike acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~b~~a)19 and 20] will be followed. The occurrence of a failed matrix spike acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11]. The lack of sufficient sample aliquot size to perform a replicate analysis should be noted in the laboratory report.

c) The activity of the laboratory control sample and matrix spike analyte(s) shall be greater than ten times and less than one hundred times the a priori detection limit.

d) The laboratory standards used to prepare the laboratory control sample and matrix spike shall be from a source independent of the laboratory standards used for instrument calibration.

e) Where a radiochemical method, other than gamma spectroscopy, has more than one reportable analyte isotope (e.g. isotopic uranium: U-234, -235, and -238) only one of the analyte isotopes need be included in the laboratory control or matrix spike sample at the indicated activity level. However, where more than one analyte isotope is present above the specified activity level each shall be assessed against the specified acceptance criteria.

f) Where gamma spectrometry is used to identify and quantitate more than one analyte isotope the laboratory control sample and matrix spike shall contain isotopes that represent the low (e.g. americium-241), medium (e.g. cesium-137) and high (e.g. cobalt-60) energy range of the analyzed gamma spectra. As indicated by these examples the isotopes need not exactly bracket the calibrated energy range or the range over which isotopes are identified and quantitated.

D.4.3 Test Variability/Reproducibility

a) Replicate - Shall be performed at a frequency of one per preparation batch where there is sufficient sample to do so. The results of this analysis shall be one of the quality control measures to be used to assess batch acceptance. The replicate result shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified replicate acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~b~~a)19 and 20] will be followed. The occurrence of a failed replicate acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

D.4.4 Other Quality Control Measures

a) Tracer - For those methods that utilize a tracer (i.e. internal standard) each sample result will have an associated tracer recovery calculated and reported. The tracer recovery for each sample results shall be one of the quality control measures to be used to assess the associated sample result acceptance. The tracer recovery shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified tracer recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~b~~a)19 and 20] will be followed. The occurrence of a failed tracer recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

b) Carrier - For those methods that utilize a carrier (i.e. internal standard) each sample will have an associated carrier recovery calculated and reported. The carrier recovery for each sample shall be one of the quality control measures to be used to assess the associated sample result acceptance. The carrier recovery shall be assessed against the specific acceptance criteria [see 5.10.1.2.b)18] specified in the laboratory method manual [see 5.10.1.2]. When the specified carrier recovery acceptance criteria is not met the specified corrective action and contingencies [see 5.10.1.2.~~b~~a)19 and 20] will be followed. The occurrence of a failed carrier recovery acceptance criteria and the actions taken shall be noted in the laboratory report [see 5.13.a)11].

D.4.5 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

a) ~~Initial~~ Demonstration of Capability - (section 5.10.2.1) shall be performed initially (prior to the analysis of any samples) and with a significant change in instrument type, personnel or method.

b) Proficiency Test Samples - The results of such analysis (5.4.2.j or 5.5.3.4) shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data. The providers of such proficiency test samples should conform to the requirements of ANSI N42.22.

D.4.6 Radiation Measurement System Calibration

Due to the stability and response nature of modern radiation measurement instrumentation it is not typically necessary to calibrate these systems in the day of use manner done so for some types of chemical measurement instrumentation. As well due to the nature of some radiation measurement instrumentation calibrations it may not be practical to calibrate in a day of use manner. In addition the calibration of modern radiation measurement instrumentation has significant differences from chemical measurement instrumentation. This section will address those practices that are necessary for proper calibration and those requirements of section 5.9.4.3 (Instrument Calibrations) that are not applicable to some types of radiation measurement instrumentation.

a) Calibration Curves

The requirements of 5.9.4.3.b)1 through 5.9.4.3.b)4 for the determination of the appropriate number of standards for initial calibration are not applicable to the performance of radiochemical methods. For those radiochemical methods that may require multiple standards for initial calibration (e.g. gas-proportional counting and liquid scintillation counting) the required number shall be addressed in the laboratory method manual [see 5.10.1.2.~~b)~~13] if not addressed in the method.

b) Calibration Curve Regression

The requirements of 5.9.4.3.c are not necessarily applicable for all radiochemical methods. Instead where linear regression is used to fit standard response or calibration standard results to a calibration curve the correlation coefficient shall be determined. Where non-linear regression is used to fit standard response or calibration standard results to a calibration curve the correlation coefficient should be determined.

c) Calibration Range

The requirements of 5.9.4.3.d are not applicable to the performance of radiochemical methods given the non-correlated event nature of decay counting instrumentation.

d) Calibration Verification

The Laboratory Control Sample may fill the requirements for the performance of an initial calibration and continuing calibration verification standard as specified in section 5.9.4.4.1 and 5.9.4.4.2. The calibration verification acceptance criteria shall be the same as specified for the Laboratory Control Sample.

e) Background Calibration- Background calibration measurements shall be made on a regular basis and monitored using control charts or tolerance charts to ensure that a laboratory

maintains its capability to meet required data quality objectives. These values are subtracted from the total measured activity in the determination of the sample activity

1) For gamma spectroscopy systems, background calibration measurements shall be performed on at least a monthly basis.

2) For alpha spectroscopy systems, background calibration measurements shall be performed on at least a monthly basis.

3) For gas-proportional and scintillation counters, background calibration measurements shall be performed on a day of use basis.

f) Calibration - Instrument calibration shall be performed with reference standards as defined in section D.4.9.a. The standards shall have the same general characteristics (i.e. geometry, homogeneity, density, etc.) as the associated samples.

g) The frequency of calibration shall be addressed in the laboratory method manual [see 5.10.1.2.~~b)~~13] if not addressed in the method. A specific frequency (e.g. monthly) or observations from the associated control or tolerance chart, as the basis for calibration shall be specified.

D.4.7 Method Detection Limits

Note: To be addressed in the next Chapter 5 revision.

D.4.8 Data Reduction

a) Refer to Section 5.10.6," Computers and Electronic Data Related Requirements," of this document.

b) Method Uncertainties - the laboratory shall have the ability to trace all sources of method uncertainties and their propagation to reported results. The ISO "Guide to the Expression of Uncertainty in Measurement" and/or the NIST Technical Note 1297 on "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results" should be used in this regard.

D.4.9 Quality of Standards and Reagents

a) The quality control program shall establish and maintain provisions for radionuclide standards.

1) Reference standards that are used in a radiochemical laboratory shall be obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers who participate in supplying NIST standards or NIST traceable radionuclides. Any reference standards purchased outside the United States shall be traceable back to each country's national standards laboratory. Commercial suppliers of reference standards should conform to ANSI N42.22 to assure the quality of their products.

2) Reference standards shall be accompanied with a certificate of calibration whose content is as described in ANSI N42.22 - 1995, Section 8, Certificates.

3) Laboratories should consult with the supplier if the lab's verification of the activity of the reference traceable standard indicates a noticeable deviation from the certified value. The laboratory shall not use a value other than the decay corrected certified value.

b) All reagents used shall be analytical reagent grade or better.

D.4.10 Constant and Consistent Test Conditions

a) To prevent incorrect analysis results caused by the spread of contamination among samples, the laboratory shall establish and adhere to written procedures to minimize the possibility of cross-contamination between samples.

b) Instrument performance checks - Instrument performance checks using appropriate check sources shall be performed on a regular basis and monitored with control charts or tolerance charts to ensure that the instrument is operating properly and that the calibration has not changed. The same check source used in the preparation of the tolerance chart or control chart at the time of calibration shall be used in the performance checks of the instrument. The check sources must provide adequate counting statistics for a relatively short count time and the source should be sealed or encapsulated to prevent loss of activity and contamination of the instrument and laboratory personnel. For alpha and gamma spectroscopy systems, the instrument performance checks shall include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy.

1) For gamma spectroscopy systems, the performance checks for efficiency and energy calibration shall be performed on a day of use basis along with performance checks on peak resolution.

2) For alpha spectroscopy systems, the performance check for energy calibration shall be performed on a day of use basis and the performance check for counting efficiency shall be performed on at least a monthly basis.

3) For gas-proportional and scintillation counters, the performance checks for counting efficiency shall be performed on a day of use basis.

~~**D.5 AIR TESTING**~~

~~Analyses for Air Toxics shall follow the essential quality controls for chemistry outlined in Appendix D.1. For air testing, the blank, laboratory control sample and a desorption efficiency (such as charcoal tubes) shall be used. Matrix spikes and duplicate samples~~

D.5 AIR TESTING

D.5.1 Negative and Positive Controls

a) Negative Controls

A. Method Blanks – Shall be performed at a frequency of one (1) per batch of twenty (20) environmental samples or less. The results of the method blank analysis shall be used to evaluate the contribution of the media and sample preparation procedure to the amount of analyte found in each sample. If the method blank contributes greater than 10% of the total

amount of analyte found, the source of the contamination must be investigated and measures taken to eliminate the source of contamination. If the source of the contamination is found to be ambient background, the data will be qualified in the report.

B. Field Blanks – Shall be prepared by the person collecting the sample(s) during sampling events at a frequency of at least one (1) field blank per sampling period. These blanks are treated as samples by the laboratory and the data are used to evaluate sample contamination during sample collection, transport, and storage.

C. Trip Blanks –Included in each shipment of sampling media to evaluate sample contamination during shipping.

D. Storage Blank – Prepared by the laboratory and may be added by the laboratory to each shipment of samples received to evaluate contamination during storage.

E. Back Section – If sampling trains are composed of "front" and "back" sections of sampling media, these sections shall be analyzed separately. The presence of greater than ten percent (10%) of the total amount of analyte in the "rear" section indicates significant analyte loss (breakthrough) unless otherwise documented by the laboratory. Samples with analytes that exhibit breakthrough shall be flagged as minimum amounts.

b) Positive Controls

A. Laboratory Control Sample – Shall be analyzed at a rate of at least one (1) per batch of twenty (20) or fewer samples for each analyte. If a spiking solution is not available, a calibration solution whose concentration approximates that of the samples, shall be included in each batch. If the target analyte concentrations are above the calibration midpoint, the LCS should be above the calibration midpoint. If the target analyte concentrations are below the calibration midpoint, the LCS should be below the calibration midpoint and if the target analyte concentrations vary across the entire calibration range, the sequential LCS concentrations shall vary across the entire calibration range.

B. Field Spikes – Prepared in the field evaluate analyte stability during transport and storage. Shall be prepared as required by the test method.

C. Trip Spikes - Shipped with each shipment of sampling media to evaluate analyte stability during transport and storage. Shall be prepared as required by the test method.

D. Recovery Spikes - Prepared in the laboratory by spiking sampling media with the target analyte(s) at the approximate levels expected in the test environment and exposed to the test environment in parallel with unspiked sampling media to evaluate analyte interference from other components in the test environment. Shall be prepared as required by the test method.

E. Desorption Efficiency (Recovery) – Desorption efficiencies shall be determined for each analyte on each lot of sampling media used to collect that analyte. The laboratory shall have SOPs defining these procedures.

F. Surrogates - Shall be used as required by the test method.

G. Matrix spike – Shall be used as required by the test method.

D.5.2 Analytical Variability/Reproducibility

1) Laboratory Duplicates – Shall be analyzed at a minimum of 1 in 20 samples per sample batch. Analysis duplicates shall be used ~~when feasible.~~ if laboratory duplicates are not available.

2) Field Duplicates – Shall be collected at a frequency of at least one (1) in twenty (20) samples, where possible and as specified by the test method.

3) Matrix Spike Duplicate – Used as required by the test method.

D.5.3 Method Evaluation

In order to ensure the accuracy of the reported result, the following procedures shall be in place:

1) Demonstration of Capability – (Sections 5.6.2 and 5.10.2.1) shall be performed prior to the analysis of any samples and with a significant change in instrument type, personnel, matrix, or test method.

2) Calibration – Calibration protocols specified in Section 5.9.4 shall be followed.

3) Proficiency Test Samples – The results of such analyses shall be used by the laboratory to evaluate the ability of the laboratory to produce accurate data.

D.5.4 Detection Limits

The laboratory shall utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. Detection limits shall be determined by the protocol in the mandated test method or applicable regulation, e.g., MDL. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

a) A detection limit study is not required for any component for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen, turbidity or on-line analyses.

b) The detection limit shall be initially determined for the compounds of interest in each test method in a matrix in which there are not target analytes nor interferences at a concentration that would impact the results or the detection limit must be determined in the matrix of interest (see definition of matrix).

c) The test method's quantitation limits (see 5.9.4.2.1.f) must be established and must be above the established detection limit.

d) Detection limits must be determined each time there is a significant change in the test method or instrument type.

e) It is essential that all sample processing steps of the analytical method be included in the determination of the detection limit.

f) All procedures used must be documented. Documentation must include the matrix type. All supporting data must be retained.

g) The laboratory must have established procedures to tie detection limits with quantitation limits.

D.5.5 Data Reduction

a) The procedures for data reduction, such as use of linear regression, shall be documented.

D.5.6 Quality of Standards and Reagents

a) The source of standards shall comply with 5.9.2.

b) The purity of each analyte standard and each reagent shall be documented by the laboratory through certificates of analyses from the manufacturer/vendor, manufacturer/vendor specifications, and/or independent analysis.

c) In methods where the purity of reagents is not specified, analytical reagent grade or higher quality, if available, shall be used.

D.5.7 Selectivity

a) The laboratory shall develop and document acceptance criteria for test method selectivity such as absolute and relative retention times, wavelength assignments, mass spectral library quality of match, and mass spectral tuning.

D.5.8 Constant and Consistent Test Conditions

a) In addition to D.1.8, the laboratory shall assure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

b) The laboratory shall document that all sampling equipment, containers and media used or supplied by the laboratory meet required test method criteria.

c) If supplied or used by the laboratory, procedures for field equipment decontamination shall be developed and their use documented.

d) The laboratory shall have a documented program for the calibration and verification of sampling equipment such as pumps, meter boxes, critical orifices, flow measurement devices and continuous analyzers, if these equipment are used or supplied by the laboratory.

QUALITY SYSTEMS
APPENDIX E

PERFORMANCE BASED MEASUREMENT SYSTEM

Appendix E - PERFORMANCE BASED MEASUREMENT SYSTEM

RESERVED - The information presented here is the most recent EMMC Workgroup draft, and is provided for information only.

E.1 CHECKLIST OVERVIEW

The Checklists present consensus among EPA's programs on performance "categories" that allow use of the same Checklists across the Agency's various programs/projects. The Checklists may be applied to screening and field techniques as well as traditional laboratory procedures.

Implementation of the Checklists is intended to be program-specific and a category that does not apply within a specific EPA program or project will be indicated by NA (not applicable). Criteria for a specific EPA program or project are to be filled in under the "Performance Criteria" column; e.g., an Office of Water Reference Method may specify 20% RSD or a correlation coefficient of 0.995 for the category that specifies calibration linearity, whereas an Office of Solid Waste project may specify a Measurement Quality Objective of 12% RSD or a correlation coefficient of 0.998 for this category.

For each EA program or project, the checklists are to be completed for each matrix within each medium for which performance is demonstrated.

Each completed Checklist must be retained on file at the laboratory that uses the performance-based method (PBM) or method modification and must be submitted to the appropriate regulatory authority upon request to support analysis of those samples to which the PBM or modified method was applied.

E.1.1 Header

Each page of the checklist contains six lines of header information, consisting of:

a) Date: enter the date that the checklist was completed and associated samples were collected.

b) Laboratory Name & Address: If the method is being employed by a commercial contract laboratory on behalf of one or more applicable clients, enter the name of the laboratory if possible followed by a listing of the appropriate clients from which the samples were collected).

c) Discharge Point ID, where applicable.

d) Facility Name: enter the name of the water treatment facility, system, or regulated facility or other program/project specified entity where the facility maintains an on-site analytical laboratory.

e) EPA Program & Applicable Regulation: enter the name of the Agency program or project to whom the results will be reported, or under the auspices of which the data are collected, e.g., “CAA” for Clean Air Act testing/monitoring and “SDWA” for analyses associated with the Safe Drinking Water Act.

f) Medium: enter the type of environmental sample, e.g., water--NOTE a separate checklist should be prepared for each matrix, e.g., for checklists associated with performance-based methods for SDWA, enter Drinking Water as the matrix type. As the evaluations of a performance-based method will involve matrix-specific performance measures, a separate checklist would be prepared for each matrix. The medium is the environmental sample type to which the performance-based method applies, whereas the performance category matrix, appearing in the body of the checklists refers to the specific sample type within the Medium that was spiked, e.g., for Medium hazardous waste, the checklist category Matrix may be solvent waste.

g) Analyte, Class of Analytes, or Other Measured Parameters--CAS # where available: As many methods apply to a large number of analytes, it is not practical to list every analyte in this field, as indicated on the form, the class of analytes may be listed here, i.e., volatile organics. However, if such a classification is used, a separate list of analytes and their respective Chemical Abstract Service Registry Numbers (CAS #) must be attached to the checklist.

E.1.2 EPA PBMS Checklist for Initial Demonstration of Method Performance

The Initial Demonstration of Method Performance involves multiple spikes into a defined sample matrix (e.g., wastewater, paper plant effluent), to demonstrate that the Performance-based Method meets the Program or Project Performance Criteria based on the performance of established Reference Method or based on Measurement Quality Objectives (analytical portion of the Data Quality Objectives). This exercise is patterned after the Initial Demonstration of Capability in C.1 of this appendix.

Footnote #1 indicates that a detailed narrative description of the initial demonstration procedure is to be provided.

Footnote #2 For multi-analyte methods, enter “see attachment” and attach a list or table containing the analyte-specific performance criteria from the reference method or those needed to satisfy measurement quality objectives. Complete only one of the two columns. For multi-analyte methods it is suggested that the list also contain the information for the “Results Obtained” and Performance Specification Achieved” columns.

Footnote #3 indicates that if a reference method is the source of the performance criteria, the reference method should be appropriate for its intended application and the listed criteria should be fully consistent with that reference method. The reference method name and EPA number (where applicable) should be delineated.

There are 34 numbered entries in the body of the checklist--each program will indicate the performance categories which do not pertain to the application/project, e.g., by listing as NA ("Not Applicable") for the corresponding performance criteria.

#1.- Written Method (addressing all elements in the EMMC format)

The details of the method used for analysis (and sampling, where applicable) should be described in a version of the method written in EMMC format. The EMMC method format includes the following sections: 1.0 Scope & Application; 2.0 Summary of Method; 3.0 Definitions; 4.0 Interferences; 5.0 Safety; 6.0 Equipment & Supplies; 7.0 Reagents & Standards; 8.0 Sample Collection, Preservation & Storage; 9.0 Quality Control; 10.0 Calibration & Standardization; 11.0 Procedure; 12.0 Data Analysis &

Calculations; 13.0 Method Performance; 14.0 Pollution Prevention; 15.0 Waste Management; 16.0 References; 17.0 Tables, Diagrams, Flowcharts & Validation Data. While this format may differ from that used in standard operation procedures (SOPs) in a given laboratory, the use of a consistent format is essential for the efficient and effective evaluation by inspectors, program and project managers/officers.

#2.- Title, Number and date/revision of "Reference Method" if applicable.

For example, Polychlorinated Dioxins and Furans, EPA Method 1613, Revision B, October, 1994.

#3.- Copy of the reference method, if applicable, maintained at the facility.

A copy of the reference method should be available to all laboratory personnel, however, it need not be attached to the checklist itself.

#4.- Differences between PBM and reference method attached, if applicable.

The laboratory should summarize the differences between the reference method and the performance-based method and attach this summary to the checklist. This summary should focus on significant differences in techniques (e.g., changes beyond the flexibility allowed in the reference method), not minor deviations such as the glassware used.

#5.– Concentrations of calibration standards.

The range of the concentrations of materials used to establish the relationship between the response of the measurement system and analyte concentration. This range must bracket any action, decision or regulatory limit. In addition, this range must include the concentration range for which sample results are measured and reported.

#6. ~~% RSD~~ %RSD or Slope/Correlation Coefficient of Calibration Regression.

This performance category refers to quantitative measures describing the relationship between the amount of material introduced into the measurement system and the response of the measurement system, such as an analytical instrument. A linear response is generally expected and is typically measured as either a linear regression (for inorganic analytes) or as the relative standard deviation (or coefficient of variation) of the response factors or calibration factors (for organic analytes). For example, traditional performance specifications consider any regression line with a correlation coefficient (r) of 0.995 or greater as linear. Also, for organic analytes, a relative standard deviation (RSD) of 15% or less is often considered linear (RCRA). The calibration relationship is not necessarily limited to a linear relationship. However, it should be remembered if the Program/Project Office or Officer/Managers specifies other calibration relationships, e.g., quadratic fit, more calibration standards are generally necessary to establish accurately the calibration. If applicable, a calibration curve, graphical representation of the instrument response versus the concentration of the calibration standards, should be attached.

#7.– Performance range tested (with units).

This range must reflect the actual range of sample concentrations that were tested and must include the concentration units. Since the procedures may include routine sample

dilution or concentration, the performance range may be broader than the range of the concentrations of the calibration standards.

#8.– Samples(s) used in initial demonstration have recommended preservative, where applicable. Sample(s) used in the initial demonstration should employ the recommended preservative, where applicable. Answer “yes” if the preservation in the reference method was used. If “no”, include a narrative description of the testing done to support use of the alternate preservation technique.

#9.– Samples(s) used in the initial demonstration must be within the recommended holding times, where applicable.

Unless holding time (time from when a sample is collected until analysis) has been specifically evaluated, this entry should be taken directly from the reference method, where applicable or standard table. If holding time has been evaluated, include the study description and conclusions of that evaluation here, with a reference to the specific study description. The data must be attached.

#10.– Interferences.

Enter information on any known or suspected interferences with the performance-based method. Such interferences are difficult to predict in many cases, but may be indicated by unacceptable spike recoveries in environmental matrices, especially when such recovery problems were not noted in testing a clean matrix such as reagent water. The interferences associated with the reference method are to be indicated, as well as, the effect of these interferences on the performance-based method.

#11.– Qualitative identification criteria used.

Enter all relevant criteria used for identification, including such items as retention time, spectral wavelengths and ion abundance ratios. If the instrumental techniques for these performance-based method are similar to a reference method, use the reference method as a guide when specifying identification criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter “see attached” for this item.

#12.– Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title last study number:).

Several EPA programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Where available and applicable, enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. A program/project may specify that a performance-based method be fully successful, i.e., within the PE study QC acceptance criteria. Where applicable, provide a listing of analytes for which the PE results were "not acceptable".

#13.– Analysis of external reference material.

Enter the results of analyses on reference material from a source different from that used to prepare calibration standards (if available). This performance category is especially important if Performance Evaluation Studies are not available for the analytes of interest.

#14.– Source of reference material.

Enter information, if applicable and available, for traceability of external reference materials used to verify the accuracy of the results, e.g., obtained from the National Institute of Science and Technology (NIST).

#15.– Surrogates used, if applicable.

Enter the names of the surrogate compounds used. Surrogates are often used in analysis of organic analytes. Surrogates may be added to samples prior to preparation, as a test of the entire analytical procedure. These compounds are typically brominated, fluorinated or isotopically labeled, with structural similarities to the analytes of interest. Target analytes of the method may be used as surrogates, if they can be demonstrated not to be present in the samples to be analyzed.

#16.– Concentrations of surrogates, if applicable.

Enter the concentration of surrogates once spiked into the sample (i.e., final concentration).

#17.– Recoveries of Surrogates appropriate to the proposed use, if applicable.

Enter the summary of the surrogate recovery limits; attach a detailed listing if more space is needed.

#18.– Sample Preparation.

Enter preliminary procedures, e.g., digestion, distillation and/or extraction. A detailed listing may be attached if more space is needed.

#19.– Clean-up Procedures.

Enter appropriate sample clean-up steps prior to the determinative step (instrumental analysis), e.g., GPC, copper, alumina treatment, etc.

#20.– Method Blank Results.

A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the concentrations of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte/s and concentration measured in the blank.

#21.– Matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.).

Refers to the specific sample type within the broader Medium that was spiked, e.g., for Medium: Hazardous Waste an example matrix spiked as part of the initial demonstration of method performance might be "solvent waste".

#22.– Spiking System, appropriate to the method and application.

Enter the procedure by which a known amount of analyte/s ("spike") was added to the sample matrix. This may include the solvent that is employed and the technique to be employed (e.g., permeation tube, or volumetric pipet delivery techniques spiked onto a soil sample and allowed to equilibrate 1 day, etc.). Solid matrices and air are often difficult to spike and considerable detailed narrative may be necessary to delineate the procedure. For spikes into aqueous samples generally a water miscible solvent is needed.

#23.– Spike concentrations (w/units corresponding to final sample concentration).

Enter the amount of the analyte/s ("spike") that was added to the sample matrix in terms of the final concentration in the sample.

#24.– Source of spiking material.

Enter the organization or vendor from which the spiking material was obtained or how the spiking material was prepared. This should include specific identification information, e.g., lot#, catalogue number, etc.

#25.– Number of Replicate Spikes.

The initial demonstration of method performance involves the analyses of replicate spikes into a defined sample matrix (category #21). Enter the number of such replicates. For example in the NPDES and SDWA programs, at least 4 replicates should be prepared and analyzed independently.

#26.– Precision (analyte by analyte).

Precision is a measure of agreement among individual determinations. Statistical measures of precision include standard deviation, relative standard deviation or percent difference.

#27.– Bias (analyte by analyte).

Bias refers to the systematic or persistent distortion of a measurement process which causes errors in one direction. Bias is often measured as the ratio of the measured value to the "true" value or nominal value. Bias is often (erroneously) used interchangeably with "accuracy", despite the fact that the two terms are complementary, that is, high "accuracy" implies low "bias", as well as good precision. Enter the name of the bias measure (% recovery, difference from true, etc.), and the numeric value with associated units for each analyte obtained for each analyte spiked in the initial demonstration procedure.

#28.– Detection Limit (w/units; analyte by analyte), if applicable.

A general term for the lowest concentration at which an analyte can be detected and identified. There are various approaches to establishing detection limits which include "Limit of Detection" and 'Method Detection Limit". Enter the approach used (e.g., MDL) and the analytical result with units for each analyte in the matrix (see #21).

This performance category is of importance when operating at extremely low concentrations. If the concentrations measured or the decisions to be made, e.g., action

levels, are several orders of magnitude above these concentrations, the "quantitation level" should be entered.

#29.– Confirmation of Detection Limit. if applicable.

In addition to spikes into the matrix of interest (see #21) it may be beneficial to perform the detection limit measurements in a clean matrix, e.g., laboratory pure water, air, sand, etc. Results of the spikes in the clean matrix are frequently available in the Agency's published methods. Determining MDLs in a clean matrix using the performance-based method will allow a comparison to the MDLs published in the Agency methods.

This performance category is of importance when operating at extremely low concentrations. If the concentrations measured or the decisions to be made, e.g., action levels, are several orders of magnitude above these concentrations, the "quantitation level" should be entered.

Also, the detection limit technique may specify specific procedures to verify that the obtained limit is correct, e.g., the "iterative process" detailed in the 40 CFR Part 136, Appendix B, MDL procedures.

#30.– Quantitation Limit (w/ units; analyte by analyte).

The lowest concentration at which the analyte can be reported with sufficient certainty that an unqualified numeric value is reported. Approaches to establishing quantitation limits include the Minimum Level (ML), Interim Minimum Level (IML), Practical Quantitation Level (PQL), and Limit of Quantitation (LOQ). Enter the approach used to establish the quantitation limits, and the corresponding units for each analyte appropriate to the intended application and a description of how hey were determined.

#31.– Qualitative Confirmation.

Enter all relevant criteria used for identification, including such items as: retention time; use of second chromatographic column; use of second (different) analytical technique; spectral wavelengths, ion abundance ratios. If the instrumental techniques for the performance-based method are similar to those of a reference method, use the reference method as a guide when specifying confirmation criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter “see attached” for this item.

#32.– Frequency of performance of Initial Demonstration:

Enter the frequency that the initial demonstration needs to be repeated.

#33-#34.– Other Criteria.

Enter other necessary program/project specific method performance categories.

Signatures:

The printed name, signature and date of each analyst involved in the initial demonstration of method performance is to be provided at the bottom of the checklist sheet.

E.1.3 EPA PBMS Checklist for Continuing Demonstration of Capability:

The process by which a laboratory documents that its previously established performance of an analytical procedure continues to meet performance specifications as delineated in this checklist.

#1.– Method Blank Result.

A clean matrix (i.e., does not contain the analytes of interest) that is carried through the entire analytical procedure, including all sample handling, preparation, extraction, digestion, cleanup and instrumental procedures. The volume or weight of the blank should be the same as that used for sample analyses. The method blank is used to evaluate the levels of analytes that may be introduced into the samples as a result of background contamination in the laboratory. Enter the analyte/s and concentration measured in the blank.

#2.- Concentrations of calibration standards used to verify working range, where applicable (include units).

The range of the concentration(s) of materials used to confirm the established relationship between the response of the measurement system and analyte concentration. This range should bracket any action, decision or regulatory limit. In addition, this range must include the concentration range for which sample results are measured and reported (when samples are measured after sample dilution/concentration). Enter the concentrations of the calibration standards.

#3.- Calibration Verification.

A means of confirming that the previously determined calibration relationship still holds. This process typically involves the analyses of two standards with concentrations which bracket the concentration(s) measured in the sample/s. Enter the procedure to be used to verify the calibration and the results obtained for each analyte.

#4.– Laboratory Control Sample.

An analytical standard carried through all aspects of the analytical method, e.g., digestions, distillations and determinative steps/instrumentation. It is generally used to assess the performance of all of the measurement system independent of the challenges of the sample matrix.

#5.– External QC sample (where applicable).

Enter the results of analyses for reference material (e.g., quality control samples/ampoules) from a source different from that used to prepare calibration standards (where applicable). Enter the concentration, as well as, the source of this material. This performance category is of particular importance if Performance Evaluation (PE) studies are not available for the analytes of interest.

#6.– Performance Evaluation Studies performed for analytes of interest, where available (last study sponsor and title last study number:).

Several EPA programs conduct periodic performance evaluation (PE) studies. Organizations outside of the Agency also may conduct such studies. Where available and applicable, enter the sponsor, title, and date of the most recent study in which the performance-based method was applied to the matrix of interest. A program/project may specify that a performance-based method be fully successful, i.e., within the PE study QC acceptance criteria.

#7.- List of analytes for which results were “not acceptable” in PE study where available and applicable..

#8.- Surrogates used, if applicable.

Enter the names of the surrogate compounds used. Surrogates are often used in analysis of organic analytes. Surrogates may be added to samples prior to preparation, as a test of the entire analytical procedure. These compounds are typically brominated, fluorinated or isotopically labeled, with structural similarities to the analytes of interest. Target analytes of the method may be used as surrogates, if they can be demonstrated not to be present in the samples to be analyzed.

#9.- Concentration of surrogates, if applicable.

Enter the concentration of surrogates once spiked into the sample (i.e., final concentration), with units.

#10.- Recoveries of Surrogates appropriate to the proposed use (if applicable).

Enter the summary of the surrogate recovery limits and attached a detailed listing (each surrogate compound), if more space is needed.

#11.- Matrix (reagent water, drinking water, sand, loam, clay, waste solid, ambient air, etc.).

Refers to the specific sample type within the broader “Medium” that was spiked, e.g., for Medium: Waste an example matrix, spiked as part of the initial demonstration of method performance, might be solvent waste.

#12.- Matrix Spike Compounds.

Enter the analytes spiked. In preparing a matrix spike, a known amount of analyte is added to an aliquot of a real-world sample matrix. This aliquot is analyzed to help evaluate the effects of the sample matrix on the analytical procedure. Matrix spike results are typically used to calculate recovery of analytes as a measure of bias for that matrix.

#13.– Matrix Spike Concentrations (w/units corresponding to final sample concentration).

Enter the amount of the analyte/s or "spike" that was added to the sample matrix in terms of the final concentration in the sample.

#14.- Recovery of Matrix Spike (w/units).

The ratio of the standard deviation of a series of at least three measurements to the mean of the measurements. This value is often expressed as a percentage of the mean.

Note: Some programs/projects have utilized matrix spike duplicates (a separate duplicate of the matrix spike) to help verify the matrix spike result and to provide precision data for analytes which are not found in real-world samples, since duplicates of non-detects provides little information concerning the precision of the method. See Item # 19.

#15.- Qualitative identification criteria used.

Enter all relevant criteria used for identification, including such items as retention times, spectral wavelengths, and ion abundance ratios. If the instrumental techniques for the performance-based method are similar to a reference method, use the reference method as a guide when specifying identification criteria. If the list of criteria is lengthy, attach it on a separate sheet, and enter “see attached” for this item.

#16.- Precision (analyte by analyte).

#17-18.- Other category.

Enter other necessary program/project specific method performance categories.

Signatures:

The printed name, signature and date of each analyst involved in the initial demonstration of method performance is to be provided at the bottom of the checklist sheet.

EPA Performance-Based Measurement System
Certification Statement

Date: Page __of __
Laboratory Name & Address
Facility Name:
Discharge Point ID, where applicable:
EPA Program and Applicable Regulation:
Medium:
(i.e., water, soil, air, waste solid, leachate, sludge, other)
Analyte, Class of Analytes or Measured Parameters (CAS # where available)
(i.e , barium, trace metals, benzene, volatile organics, etc.)

We, the undersigned, CERTIFY that:

1. The methods in use at this facility for the analyses of samples for the programs of the U.S. Environmental Protection Agency have met the Initial and any required Continuing Demonstration of Method Performance Criteria specified under the Performance-Based Measurement System.

2. A copy of the Performance-Based Method, written in EMMC format, and copies of the reference method and laboratory-specific SOPs are available for all personnel on-site.

3. The data and checklists associated with the initial and continuing demonstration of method performance are true, accurate, complete and self-explanatory (1).

4. All raw data (including a copy of this certification form) necessary to reconstruct and validate these performance related analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized inspectors.

_____	_____	_____
Facility Manager's Name and Title	Signature	Date
_____	_____	_____
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed when the performance-based method is originally certified, each time a continuing demonstration of method performance is documented, and whenever a change of personnel involves the Facility Manager or the Quality Assurance Officer.

(1) True: Consistent with supporting data.

Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.

Complete: Includes the results of all supporting performance testing.

Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

EPA PBMS
Checklist for Initial Demonstration of Method Performance

Provide a checklist for each matrix included in the demonstration.

Date: **Page __of __**
Laboratory Name & Address:
Facility Name:
Discharge Point ID, where applicable:
EPA Program and Applicable Regulation:
Medium:
(i.e., water, soil, air, waste solid, leachate, sludge, other)
Analyte, Class of Analytes or Other Measured Parameters (CAS #, where available):
(i.e., barium, trace metals, benzene, volatile organics, etc.)

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
1. Written method (addressing all elements in the EMMC format) attached				
2. Title, number and date/rev. of "reference method", if applicable ***(3)***				
3. Copy of the reference method, if applicable, maintained at facility				
4. Differences between PBM and reference method (if applicable) attached				
5. Concentrations of calibration standards				
6. %RSD or slope/correlation coefficient of calibration regression				
7. Performance range tested (with units)				
8. Sample(s) used in initial demonstration have recommended preservative, where applicable.				
9. Samples(s) used in initial demonstration met recommended holding times, where applicable				
10. Interferences				
11. Qualitative identification criteria used				

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
12. Performance Evaluation studies performed for analytes of interest, where available: Last study sponsor and title: Last study number:				
13. Analysis of external reference material Last study sponsor and title: Last study number: List of analytes with “not acceptable” results:				
14. Source of reference material				
15. Surrogates used, if applicable				
16. Concentrations of surrogates, if applicable				
17. Recoveries of Surrogates appropriate to the proposed use, if applicable				
18. Sample preparation				
19. Clean-up procedures				
20. Method Blank Result				
21. Matrix (reagent water, drinking water, sand, waste solid, ambient air, etc.)				
22. Spiking system, appropriate to method and application				
23. Spike concentrations (w/ units corresponding to final sample concentration)				
24. Source of spiking material				
25. Number of replicate spikes				
26. Precision (analyte by analyte)				
27. Bias (analyte by analyte)				
28. Detection Limit (w/ units; analyte by analyte)				
29. Confirmation of Detection Limit, if applicable				
30. Quantitation Limit (w/ units: analyte by analyte)				

Initial Demonstration of Method Performance *(1)*				
Category	**Performance Criteria *(2)* Based on**		**Results Obtained**	**Perf. Spec. Achieved (✓)**
	Reference Method	**Measurement Quality Objective**		
31. Qualitative Confirmation				
32. Frequency of performance of the Initial Demonstration				
33. Other criterion (specify)				
34. Other criterion (specify)				

[1] **Provide a detailed narrative description of the initial demonstration.**

[2] **For multi-analyte methods, enter "see attachment" and attach a list or table containing the analyte-specific performance criteria from the reference method or those needed to satisfy measurement quality objectives.**

[3] **If a reference method is the source of the performance criteria, the reference method should be appropriate to the required application, and the listed criteria should be fully consistent with that reference method.**

Name and signature of each analyst involved in the initial demonstration of method performance (includes all steps in the proposed method/modification):

_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**

The certification above must accompany this form each time it is submitted.

EPA PBMS
Checklist for Continuing Demonstration of Method Performance

Date: **Page __of __**
Facility Name:
Laboratory Name & Address:
Discharge Point ID, where applicable:
EPA Program and Applicable Regulation:
Medium:
(i.e.,water, soil, air, waste solid, leachate, sludge, other)
Analyte, Class of Analytes or Measured Parameters (CAS # where available)
(i.e., barium, trace metals, benzene, volatile organics, etc.)

Continuing Demonstration of Method Performance				
Category	Required Frequency	Specific Performance Criteria	Results Obtained	Perf. Spec. Achieved (✓)
1. Method blank result (taken through all steps in the procedure)				
2. Concentrations of calibration standards used to verify working range (with units), where applicable				
3. Calibration verification				
4. Laboratory Control Sample				
5. External QC sample (where available)				
6. Performance evaluation (PE) studies, if applicable Last study sponsor and title: Last study number:				
7. List analytes for which results were "not acceptable" in PE study	----	----	----	----
8. Surrogates used, if applicable				
9. Concentration of Surrogates, if applicable				
10. Recovery of Surrogates (acceptance range for multianalyte methods), if applicable				
11. Matrix				
12. Matrix spike compounds				
13. Concentration of Matrix spike compounds				
14. Recoveries of Matrix spike compounds				
15. Qualitative identification criteria used				
16. Precision (analyte by analyte)				
17. Other category (specify)				
18. Other category (specify)				

EPA PBMS
Checklist for Continuing Demonstration of Method Performance

Date: **Page __of __**

Facility Name:

Discharge Point ID, where applicable:

EPA Program and Applicable Regulation:

Medium:
(i.e. water, soil, air, waste solid, leachate, sludge, other)

Analyte, Class of Analytes or Measureand (CAS # where available)
(i.e. barium, trace metals, benzene, volatile organics, etc.)

Name and signature of each analyst involved in continuing demonstration of method performance (includes all steps in the proposed method/modification):

_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**
_____	_____	_____
Name	**Signature**	**Date**

The certification above must accompany this form each time it is submitted.

QUALITY SYSTEMS
APPENDIX F

LISTING OF PROCEDURES

Appendix F - LISTING OF PROCEDURES

The information is provided for information only.

F. 1 Overview

The procedures listed in this appendix are extracted from Chapter 5 excluding the appendices. This appendix is guidance for assessing quality system compliance with chapter 5 requirements. Each laboratory+s quality system may be unique based on its business processes, quality policy, objectives and customer requirements. Therefore, the laboratory+s procedures are not expected to be named as presented in this listing. Also, the laboratory may find it necessary to have additional quality system procedures to effectively operate its laboratory business. The laboratory has the flexibility to design a system that meets their needs. Quality system procedures may be combined, split into work instructions, or even listed as process flow charts as long as it can demonstrate compliance with the NELAC standard.

Those using this appendix are expected to refer to the context of Chapter 5 from which these citations are taken to assure that the context is understood and complies with the standard.

F.2 Listing

Policy/Procedure	**NELAC Reference**
1) Client confidentiality and proprietary rights	5.4.2i & 5.5.2r
2) Management Quality System Review	5.5.3.2
3) Contract review, design control & quality planning	5.5.2i
4) Document Control	Not Referenced
5) Analytical subcontracting	5.14
6) Purchasing, receiving and storage of technical supplies	5.15 & 5.10.5
7) Sample acceptance Policy	5.11.2 & 5.11.3
8) Sample handling, transport, and storage	5.10.1
9) Sample disposal	5.11.5
10)Sample identification and traceability	5.11.1a-c
11)Signature Authority	5.4.2h
12)Sample Preparation	5.10.1a & 5.10.2a
13)Data review	5.10.4b & 5.5.3.5a & 5.5.2s
14)Equipment operations	5.10.1
15)Equipment maintenance	5.8b & 5.5.2m
16)Equipment Verification and calibration program	5.5.2m & 5.9.2b

Policy/Procedure	**NELAC Reference**
17)Acceptable permitting departures from documented policies, procedures, and specifications	5.5.2p
18)Corrective action	5.5.2o & 5.5.3.5a
19)Complaint handling	5.5.2q & 5.16
20)Preventive action	Not Referenced
21)Reporting analytical results	5.5.2u
22)Electronic reporting	5.13f
23)Record retention	5.12 & 5.5.2d
24)Records transfer	5.12.2f
25)Internal audits	5.5.2s
26)Training - Personnel experience review	5.5.2t
27)Subsampling	5.10.3
28)Developing acceptance criteria when no method or regulatory requirement exists	5.5.4c
29)Data integrity	5.10.6c
30)Data security	5.10.6e
31)Glassware cleaning	D.1.8b
* Not Referenced: Procedures that are not listed in Chapter 5 but are fundamental quality system procedures.	

QUALITY SYSTEMS
APPENDIX G

LISTING OF RECORDS AND DOCUMENTATION

Appendix G - LISTING OF RECORDS AND DOCUMENTATION:

The information is provided for information only.

G.1 Overview

The documentation requirements listed in this appendix are extracted from Chapter 5 excluding the appendices. This appendix is guidance for assessing quality system compliance with chapter 5 requirements. Each laboratory's records system may be unique based on its business processes, quality policy, objectives and customer requirements. Also, the laboratory may find it necessary to have additional documentation requirements to effectively operate its laboratory business. The laboratory has the flexibility to design a system that meets their needs. While this extraction is believed to be complete the responsibility of the laboratory to meet documentation requirements are not relieved by the failure for such requirements to be included in this appendix.

Those using this appendix are expected to refer to the context of Chapter 5 from which these citations are taken to assure that the context is understood and complies with the standard.

G. 2 Listing

This is a cross-reference of section 5.12.3.1 through 5.12.3.4, (July 2, 1998 version) with the explicit requirements throughout the chapter, except appendicees. Those items under "Miscellaneous" below don't explicitly map to the categories in 5.12.3.1 through 5.12.3.4.

G.2.1 Miscellaneous:

5.5.2	The Quality Manual shall list on the title page: a document title; the laboratory's full name and address; the name, address (if different from above), and telephone number of individual(s) responsible for the laboratory; the name of the quality assurance officer (however named); the identification of all major organizational units which are to be covered by this quality manual and the effective date of the version;
5.5.2(a)	The quality manual and related quality documentation shall also contain … a quality policy statement, including objectives and commitments, by top management;
5.5.2(b)	The quality manual and related quality documentation shall also contain … the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;
5.5.2(c)	The quality manual and related quality documentation shall also contain … the relationship between management, technical operations, support services and the quality system;
5.5.2(f)	The quality manual and related quality documentation shall also contain … identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed concurrence, (with appropriate titles) of all responsible parties including the QA officer, technical director, and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager;

5.5.2(i)	The quality manual and related quality documentation shall also contain … mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;
5.5.2(v)	The quality manual and related quality documentation shall also contain … a Table of Contents, and applicable lists of references and glossaries, and appendices.
5.6.2(d)	Documenting all analytical and operational activities of the laboratory;
5.10.2.1(c)	In all cases, the appropriate forms such as the Certification Statement (Appendix C) or standard performance checklists (see Appendix E) must be completed and retained by the laboratory to be made available upon request. All associated supporting data necessary to reproduce the analytical results summarized in the checklists must be retained by the laboratory.
5.12.1	All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification shall be documented.
5.12.2(d)	The laboratory shall establish a record management system for control of laboratory notebooks; instrument logbooks; standards logbooks; and records for data reduction, validation storage and reporting;
5.15(c)	The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for tests.

G. 2.2 Cross-reference of section 5.12.3.1, "Sample Handling", (July 2, 1998 version) with the explicit requirements throughout the chapter, except appendicees.

Sample preservation including appropriateness of sample container and compliance with holding time requirement;

Sample identification, receipt, acceptance or rejection and log-in;

5.11.2	The laboratory shall have a written sample acceptance policy that clearly outlines the circumstances under which samples will be accepted. Data from any samples which do not meet the following criteria must be flagged in an unambiguous manner clearly defining the nature and substance of the variation. This sample acceptance policy shall be made available to sample collection personnel and shall include, but is not limited to, the following areas of concern:
5.11.2(a)	Proper, full, and complete documentation, which shall include sample identification, the location, date and time of collection, collector's name, preservation type, sample type and any special remarks concerning the sample;
5.11.2(f)	Procedures to be used when samples which show signs of damage or contamination.
5.11.3(a)	Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition as prescribed in the relevant test method, shall be recorded. All items specified in 5.11.2 above shall be checked.
5.11.3(b)	The results of all checks shall be recorded.

Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records;

5.5.2(k)	The quality manual and related quality documentation shall also contain … procedures for handling submitted samples;
5.11.1(a)	The laboratory shall have a documented system for uniquely identifying the items to be tested, to ensure that there can be no confusion regarding the identity of such items at any time. This system shall include identification for all samples, subsamples and subsequent extracts and/or digestates. The laboratory shall assign a unique identification (ID) code to each sample container received in the laboratory. The use of container shape, size or other physical characteristic, such as amber glass, or purple top, is not an acceptable means of identifying the sample.
5.11.1(d)	The laboratory ID code shall be entered into the laboratory records (see 5.11.3.d) and shall be the link that associates the sample with related laboratory activities such as sample preparation or calibration.
5.11.3(d)	The laboratory shall utilize a permanent chronological record such as a log book or electronic database to document receipt of all sample containers.
5.11.3(d)(2)	During the log in process, the following information must be unequivocally linked to the log record or included as a part of the log. If such information is recorded/documented elsewhere, the records shall be part of the laboratory's permanent records, easily retrievable upon request and readily available to individuals who will process the sample. Note: the placement of the laboratory ID number on the sample container is not considered a permanent record.
5.11.3(f)	A complete chain of custody record (Section 5.12.4), if utilized, shall be maintained.
5.11.4	The laboratory shall have documented procedures and appropriate facilities to avoid deterioration, contamination, or damage to the sample during storage, handling, preparation, and testing; any relevant instructions provided with the item shall be followed. Where items have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored and recorded where necessary.
5.12.4.2	In addition to the information specified in 5.11.1.a and 5.11.1.b, tracking records shall include, by direct entry or linkage to other records: a) Time of day and calendar date of each transfer or handling procedure; b) Signatures of all personnel who physically handle the sample(s); c) All information necessary to produce unequivocal, accurate records that document the laboratory activities associated with sample receipt, preparation, analysis and reporting; and d) Common carrier documents.
5.14	a) The laboratory shall advise the client in writing of its intention to sub-contract any portion of the testing to another party. b) Where a laboratory sub-contracts any part of the testing covered under NELAP, this work shall be placed with a laboratory accredited under NELAP for the tests to be performed. c) The laboratory shall retain records demonstrating that the above requirements have been met.

Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

Sample analysis;

Standard and reagent origin, receipt, preparation, and use;

5.9.2(b)	Calibration certificates shall when available indicate the traceability to national standards of measurement and shall provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with an identified metrological specification. The laboratory shall maintain records of all such certifications.
5.9.2(c)	Where traceability to national standards of measurement is not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons, proficiency testing, or independent analysis.
5.10.5	Documented procedures shall exist for the purchase, reception and storage of consumable materials used for the technical operations of the laboratory.
5.10.5(a)	The laboratory shall retain records for all standards including the manufacturer/vendor, the manufacturer's Certificate of Analysis or purity (if supplied), the date of receipt, recommended storage conditions, and an expiration date after which the material shall not be used unless it is verified by the laboratory.
5.10.5(c)	Detailed records shall be maintained on reagent and standard preparation. These records shall indicate traceability to purchased stocks or neat compounds, reference to the method of preparation, date of preparation, expiration date and preparer's initials.

Equipment receipt, use, specification, operating conditions and preventative maintenance;

5.5.2(l)	The quality manual and related quality documentation shall also contain … reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;
5.5.2(m)	The quality manual and related quality documentation shall also contain … reference to procedures for calibration, verification and maintenance of equipment;
5.7.1(d)	In instances where monitoring or control of any of the above mentioned items are specified in a test method or by regulation, the laboratory shall meet and document adherence to the laboratory facility requirements.
5.8(b)	All equipment shall be properly maintained, inspected and cleaned. Maintenance procedures shall be documented.

5.8(e)	Records shall be maintained of each major item of equipment and all reference materials significant to the tests performed. These records shall include documentation on all routine and non-routine maintenance activities and reference material verifications. The records shall include: 1) the name of the item of equipment; 2) the manufacturer's name, type identification, and serial number or other unique identification; 3) date received and date placed in service (if available); 4) current location, where appropriate; 5) if available, condition when received (e.g. new, used, reconditioned); 6) copy of the manufacturer's instructions, where available; 7) dates and results of calibrations and/or verifications and date of the next calibration and/or verification; 8) details of maintenance carried out to date and planned for the future; and 9) history of any damage, malfunction, modification or repair.
5.9.4.2.1(a)	maintained in proper working order. The records of all activities including service calls shall be kept.
5.9.4.2.2	The sterilization temperature and pressure of each run must be documented by the use of appropriate chemical or biological sterilization indicators. Autoclave tape may be used to indicate by color change that a load has been processed, but not to demonstrate completion of an acceptable sterilization cycle. Demonstration of sterilization may be provided by a continuous temperature recording or with the use of spore strips.
5.10.1(a)	The laboratory shall have documented instructions on the use and operation of all relevant equipment, on the handling and preparation of samples and for calibration and/or testing, where the absence of such instructions could jeopardize the calibrations or tests.
5.10.1(b)	All instructions, standards, manuals and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.

Calibration criteria, frequency and acceptance criteria;

5.5.2(g)	The quality manual and related quality documentation shall also contain … the laboratory's procedures for achieving traceability of measurements;
5.9.4.1(b)	Sufficient information shall be recorded to permit reconstruction of the calibration.
5.9.4.1(c)	Criteria for the acceptance of a calibration procedure, such as calibration curves and concentration (titer) determinations of titrants, shall be established. If applicable, the method specified criteria shall be met.
5.9.4.2.1(b)(2)	The laboratory shall prepare a deviation curve and correct all measurements for the deviation. All measurements shall be recorded and maintained.

Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;

5.12	The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. The system shall produce unequivocal, accurate records which document all laboratory activities. The laboratory shall retain on record all original observations, calculations and derived data, calibration records and a copy of the test report for an appropriate period.

Method performance criteria including expected quality control requirements;

5.5.2(j)	The quality manual and related quality documentation shall also contain … reference to the calibration and/or verification test procedures used;

Quality control protocols and assessment;

Electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries;

5.10.6(b)	computer software is documented and adequate for use;
5.10.6(c)	procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission and data processing;
5.10.6(e)	it establishes and implements appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records.
5.12.2(c)	Records that are stored or generated by computers or personal computers (PCS) shall have hard copy or write-protected backup copies.

All automated sample handling systems; and

Disposal of hazardous samples including the date of sample or subsample disposal and name of the responsible person.

5.11.5	The laboratory shall have standard operating procedures for the disposal of samples, digestates, leachates and extracts or other sample preparation products.
5.12.4.5(b)	All conditions of disposal and all correspondence between all parties concerning the final disposition of the physical sample shall be recorded and retained.
5.12.4.5(c)	Records shall indicate the date of disposal, the nature of disposal (such as sample depleted, sample disposed in hazardous waste facility, or sample returned to client), and the name of the individual who performed the task.

This is a cross-reference of section 5.12.3.2, "Laboratory Support Activities ", (July 2, 1998 version) with the explicit requirements throughout the chapter, except appendicees.

All original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);

A written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;

Copies of final reports;

5.5.2(u)	The quality manual and related quality documentation shall also contain … reference to procedures for reporting analytical results; and

Archived standard operating procedures;

5.5.1(a)	The elements of this quality system shall be documented in the organization's quality manual.
5.5.1(c)	The laboratory shall define and document its policies and objectives for, and its commitment to accepted laboratory practices and quality of testing services.
5.5.1(d)	The laboratory management shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood, and implemented by all laboratory personnel concerned.
5.5.2(d)	The quality manual and related quality documentation shall also contain … procedures to ensure that all records required under this Chapter are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;
5.5.2(h)	The quality manual and related quality documentation shall also contain … a list of all test methods under which the laboratory performs its accredited testing;
5.10.1.1	Laboratories shall maintain standard operating procedures that accurately reflect all phases of current laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods. a) These documents, for example, may be equipment manuals provided by the manufacturer, or internally written documents. b) The test methods may be copies of published methods as long as any changes in the methods are documented and included in the methods manual (see 5.10.1.2). c) Copies of all SOPs shall be accessible to all personnel. d) The SOPs shall be organized . e) Each SOP shall clearly indicate the effective date of the document, the revision number and the signature(s) of the approving authority.
5.10.1.2(a)	The laboratory shall have and maintain an in-house methods manual(s) for each accredited analyte or test method.
5.10.2(a)(2)	Where test methods are employed that are not required, as in the Performance Based Measurement System approach, the methods shall be fully documented and validated (see 5.10.2.1), and be available to the client and other recipients of the relevant reports.
5.10.3	Where sampling (as in obtaining sample aliquots from a submitted sample) is carried out as part of the test method, the laboratory shall use documented procedures and appropriate techniques to obtain representative subsamples.

Correspondence relating to laboratory activities for a specific project;

5.4.2(i)	The quality assurance officer (and/or his/her designees) shall … have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights (this may not apply to in-house laboratories);
5.5.2(q)	The quality manual and related quality documentation shall also contain … procedures for dealing with complaints;
5.5.2(r)	The quality manual and related quality documentation shall also contain … procedures for protecting confidentiality (including national security concerns), and proprietary rights;
5.5.3.1	Where the audit findings cast doubt on the correctness or validity of the laboratory's calibrations or test results, the laboratory shall take immediate corrective action and shall immediately notify, in writing, any client whose work may have been affected.
5.11.3(c)	Where there is any doubt as to the item's suitability for testing, where the sample does not conform to the description provided, or where the test required is not fully specified, the laboratory should consult the client for further instruction before proceeding. The laboratory shall establish whether the sample has received all necessary preparation, or whether the client requires preparation to be undertaken or arranged by the laboratory. If the sample does not meet the sample receipt acceptance criteria listed in 5.11.3.a, 5.11.3.b or 5.11.3.c, the laboratory shall either: 1) Retain correspondence and/or records of conversations concerning the final disposition of rejected samples; or 2) Fully document any decision to proceed with the analysis of samples not meeting acceptance criteria.
5.11.3(e)	All documentation, such as memos or transmittal forms, that is transmitted to the laboratory by the sample transmitter shall be retained.
5.13(e)	The laboratory shall notify clients promptly, in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any calibration certificate, test report or test certificate or amendment to a report or certificate.
5.13(f)	The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure that the requirements of this Standard are met and that confidentiality is preserved.
5.16	The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. Where a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this Standard or otherwise concerning the quality of the laboratory's calibrations or tests, the laboratory shall ensure that those areas of activity and responsibility involved are promptly audited in accordance with Section 5.5.3.1. Records of the complaint and subsequent actions shall be maintained.

All corrective action reports, audits and audit responses;

5.5.2(o)	The quality manual and related quality documentation shall also contain … procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;

5.5.2(p)	The quality manual and related quality documentation shall also contain … the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;
5.5.2(s)	The quality manual and related quality documentation shall also contain … procedures for audits and data review;
5.5.3.3	All audit and review findings and any corrective actions that arise from them shall be documented.

Proficiency test results and raw data; and

5.5.2(n)	The quality manual and related quality documentation shall also contain … reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;
5.6.2(b)	Ensuring that all technical laboratory staff have demonstrated initial and ongoing proficiency in the activities for which they are responsible. Such demonstration shall be documented;

Data review and cross checking.

5.6.2(g)	Documenting the quality of all data reported by the laboratory.
5.10.4(a)	The laboratory shall establish Standard Operating Procedures to ensure that the reported data is free from transcription and calculation errors.
5.10.4(b)	The laboratory shall establish a Standard Operating Procedures to ensure that all quality control measures are reviewed, and evaluated before data are reported.

This is a cross-reference of section 5.12.3.3, "Analytical Records", (July 2, 1998 version) with the explicit requirements throughout the chapter, except appendicees.

Laboratory sample ID code;

Date of analysis;

Instrumentation identification and instrument operating conditions/parameters (or reference to such data);

Analysis type;

All manual calculations ; and

Analyst's or operator's initials/signature.

This is a cross-reference of section 5.12.3.4, "Administrative Records", (July 2, 1998 version) with the explicit requirements throughout the chapter, except appendicees.

Personnel qualifications, experience and training records;

5.4.2(d)	specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests; Such documentation shall include: 1) a clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured and 2) job descriptions for all positions.
5.4.2(f)	The technical director(s) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented.
5.4.2(g)(4)	The quality assurance officer (and/or his/her designees) shall … have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system as defined under NELAC;
5.5.2(e)	The quality manual and related quality documentation shall also contain … job descriptions of key staff and reference to the job descriptions of other staff;
5.5.2(t)	The quality manual and related quality documentation shall also contain … processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and/or receive any needed training;
5.6.2(c)(1)	Evidence must be on file that demonstrates that each employee has read, understood, and is using the latest version of the laboratory's in-house quality documentation, which relates to his/her job responsibilities.

5.6.2.(c)(2)	Training courses or workshops on specific equipment, analytical techniques or laboratory procedures shall all be documented.
5.6.2.(c)(3)	Analyst training shall be considered up to date if an employee file contains a certification that technical personnel have read, understood and agreed to perform the most recent version of the test method (the approved method or standard operating procedure) and documentation of continued proficiency by at least one of the following once per year:

Initial and continuing demonstration of proficiency for each analyst; and

5.6.3	Records on the relevant qualifications, training, skills and experience of the technical personnel shall be maintained by the laboratory [see 5.6.2.c)], including records on demonstrated proficiency for each laboratory test method, such as the criteria outlined in 5.10.2.1 for chemical testing.

A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.